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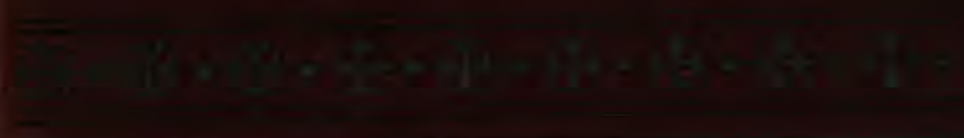
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# ASSAYING

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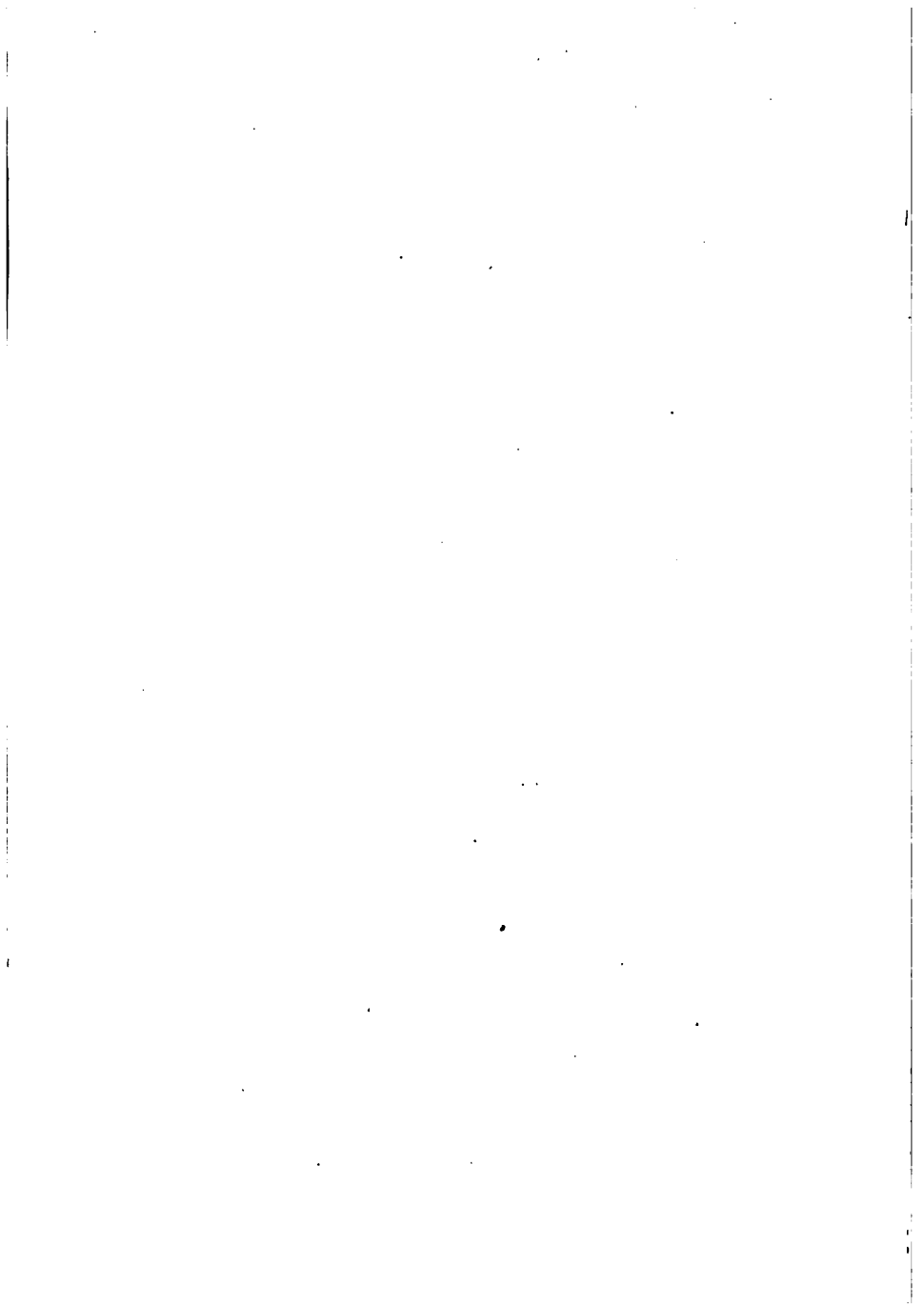
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# ASSAYING

IN

## THREE PARTS.

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PART 1ST.—GOLD AND SILVER ORES; PART 2D.—GOLD AND SILVER  
BULLION; PART 3D.—LEAD, COPPER, TIN, MERCURY,  
ZINC, NICKEL AND COBALT, CHROMIUM,  
BISMUTH, ARSENIC, ANTIMONY,  
SULPHUR, SALT.

---

By C. H. AARON, METALLURGIST,

AUTHOR OF

"TESTING AND WORKING SILVER ORES," "LEACHING GOLD AND SILVER ORES."

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PARTS II, AND III,  
THIRD EDITION.

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TO

**The Mining and Scientific Press,**

OF

**SAN FRANCISCO, CAL.,**

**THE STEADFAST FRIEND OF TRUTH AND PROGRESS  
—AN HONEST PAPER,**

**THIS BOOK IS RESPECTFULLY DEDICATED  
BY THE AUTHOR.**





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## PART II.

### GOLD AND SILVER BULLION.

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IN the assay of bullion, as in that of ore, the first step is to obtain a correct sample, and for this reason it is desirable that the bars of ingots should be made in the establishment in which they are assayed; it is not often that an assayer will place his stamp on a bar without knowing to a certainty that the bar is what it purports to be. Moreover, the best sample is one that is taken from the molten metal, though this is not practised in the case of gold bullion, and it often happens that a lot of silver lead in bars, which in this country is called "base bullion," must be sampled without melting for the purpose. Silver bullion is sampled when melted, before casting. Silver lead is so sampled when practicable. Gold bullion, or base bullion in bars, is sampled by chipping or boring.\*

Gold bullion is assayed by inquartation, involving cupellation, and parting; silver bullion by cupellation

---

\* Silver bars are also chipped or bored when the metal has been ladled into the moulds from a refining hearth. Pigs of lead containing precious metal are best sampled by drilling, or by means of a hollow punch, which is driven half through from top and bottom. The samples from a number of pigs are melted together at low heat under borax, and cast into a small bar. This bar is then cut in two, and slices are taken from top to bottom for the assays, of which several are made for an average.

or by the *humid method*; silver lead by cupellation, sometimes preceded by scorification.

Bars of bullion are called gold, doré, silver, or base. The first consist almost entirely of gold, with a little silver and sometimes a very little base metal. They are stamped with the gold fineness and value only, the silver being allowed for in the market price. The second contain a large proportion of gold, a considerable proportion of silver, and sometimes a little base metal. Silver bars are such as consist mainly of silver with little or no gold, nor an excess of base metal. Base bars contain a large proportion of base metal, usually lead or copper. Doré, silver and base bars are stamped with the fineness and value of the gold and of the silver. Gold bars containing more than an insignificant percentage of base metal, although not enough to degrade them to the rank of base bars, are marked B, or Base, in addition to the gold fineness and value. Such are the rules of commercial assaying on the Pacific Coast.

In order to extend the business of ore assaying to bullion assaying also, certain additional apparatus must be provided, as follows:—

## APPARATUS.

---

**MELTING FURNACE.**—A melting furnace suitable for light work may be bought ready made, and is similar to the crucible furnace for ore assays. The accompanying cut shows a complete and satisfactory portable furnace weighing 100 lbs. It is made of clay in one piece, and is securely bound with steel doors, asbestos lined. Will take muffle 6x12x4 inches; price with one muffle \$20; extra muffle, \$1.00;

extra grate, \$1.00. For heavy work, such as casting large bars, it is best to build a furnace of masonry, and, though not customary, it is advantageous to have it sunk in the ground to within about a foot of the top.

Such furnace would be suitable for a No. 30 or 35 black-lead pot, with charcoal as fuel. There need be no heavy cast-iron plate on the top, and the cover might be of sheet iron, although cast iron is better, the interior cylindrical, but better narrowed toward the top and bottom, the grate bars to rest on an iron ring supported by an offset in the masonry. The latter should be mostly of a very rough kind, as the furnace is sunk in the ground to within a foot of the top, which, besides affording great convenience in working, obviates the necessity of iron bands or stay rods to support it against the expansion caused by the heat. The greater part of the lining, 4 inches thick, to be of good clay, very slightly moistened, and beaten round a hollow wooden core, for which part of a barrel of suitable size answers very well; to be topped with a course of common bricks, and above these a flat iron ring, with lugs, which may be bolted down, or simply let in flush with the top of the furnace. The grate bars should not be in, but left free to be removed whenever desired.

The furnace is constructed, when convenient, at the base of the main smokestack, with which it is connected by a flue; otherwise a large stovepipe will answer for a chimney.

When the weight of melted metal does not exceed 100 pounds avoirdupois, the pot is lifted out of the furnace by hand, by means of the basket tongs, the mould being a platform of masonry on one side of, and level with, the furnace. The melter stands on the top of the furnace to lift, then steps to the ground, one foot lower, to pour.

If larger quantities of metal are melted at once, a lever may be employed for lifting the pot. The lever is arranged like that of a blacksmith's bellows, being supported by a rope, chain, or swivel, in such a manner as to admit of a lateral as well as a vertical movement, and at a height of not less than six feet above the furnace, the center of which is directly under the shorter arm.

From the longer arm of the lever a rope depends, from the shorter arm a link of half-inch iron long enough to reach, when drawn downward, into the cavity of the furnace, and terminating in a hook engaging in an eye-bolt which forms the pivot connecting the jaws of the tongs.

The tongs being adjusted on the pot, a ring is slipped over the handles to hold them together; the melter steadies them, while an assistant, pulling on the rope at the other end of the lever, lifts the pot out of the furnace and swings it near to the mould, when the melter pours the metal.

The hands and arms of the melter are protected by gloves, which, in some works, are elaborately made of canvas and padding. To make a glove simply take an ore sack, double it lengthwise, and sew it so as to form a narrow bag of two thicknesses of canvas into which the arm may be thrust to the shoulder. The gloves may be wetted to prevent burning, but hot articles must not be grasped with a wet glove, because the steam produced will scald the hand; yet moisture is a good protection against radiant heat while grasping the cool handles of the tongs.



In melting with charcoal, the best result is obtained, not by keeping the furnace full, but by letting nearly all the fuel burn away before refilling. A little practise will enable the melter so to manage that there shall be but little coal in the furnace when the time for pouring arrives, so that it is not in the way when seizing the pot with the basket tongs. The lifting must be performed without delay, otherwise the tongs may become red hot and bend. The feet and legs of the melter may be protected by woolen armor, or by wetting the boots and trowsers; but melters and assayers must not shrink from a little scorching.

If there is much fuel left in the furnace when the melting is ended, the grate is taken out, and the embers fall into the ash pit, where they are extinguished.

Clinkers, if formed on the sides of the furnace from the melting of the lining, or from dirty fuel, are punched off while red hot by means of an iron bar with a chisel end. If they were to be removed after cooling, the walls would be broken.

If making a bar of clean skimmed metal, it is proper to throw a little resin or powdered charcoal into the pot a few seconds before pouring; it prevents the sputtering which is often caused by the formation of base oxides. As soon as such a bar is cast, the top of it is covered with charcoal powder to prevent oxidation while solidifying. When the slag or matte is poured with the metal and covers the top of the bar, the charcoal is not required. Sputtering is sometimes caused by sulphur in the metal, in which case charcoal does not answer; the metal may then be stirred for some

time with a rod of iron and a little borax thrown on it, which is afterward skimmed off.

In a first-class office, the top of the furnace consists of a heavy plate of cast iron, sloping upward toward the flue, and provided with sliding covers. A melting furnace should have a strong draft controlled by a damper in the chimney. The flue connecting the furnace with the stack or chimney should be 10 inches or more in width and about 6 inches high, in order to admit of placing a 6-inch cupel within it for a purpose which will appear hereafter. The floor, to a width of 4 feet on the front and sides of the furnace or set of furnaces, should be paved with bricks. A furnace of which the interior is 16 inches square is large enough for the melting of 2,000 ounces of silver at once.



**BLACK-LEAD CRUCIBLES.**—Made of a mixture of clay and plumbago; used for the melting of bullion in the melting furnace; can be used from 10 to 20 times.

No.....	1	2	3	4	5
* Prices, each.....	30c	35c	40c	45c	55c
Price, covers, $\frac{1}{2}$ doz .....	\$2.00	2.25	2.25	2.50	2.50
No.....		6	8	10	12
Prices, each.....		60c	70c	80c	90c
Price, covers, $\frac{1}{2}$ doz .....		\$2.75	3.00	3.25	3.60
Numbers 14, 16, 18, 20, 25, 30, 35, 40, 45, 50, 60, 70, 80, 90, 100, 150; price, per number, 7c.					
Covers, price, per number, 3 cts.					

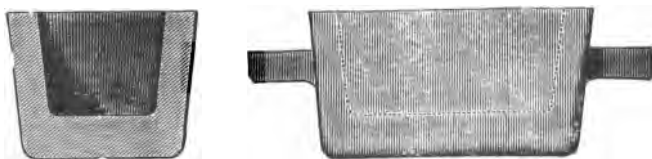
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\* Subject to market fluctuations.

**TONGS.**—Straight, bent, and basket tongs of several sizes adapted to the crucibles. The basket tongs are constructed to grasp the pot around below the bulge, or both above and below, by curved arms attached to the jaws; they are used only for the larger sizes of pots, say above No. 20. Smaller pots are seized by the rim with the straight or crooked tongs. Also bar tongs with broad, flat, and long jaws for lifting hot bars. Straight pokers for adjusting the coals, and bent for clearing the grates from below, are needed.

**SKIMMERS.**—Iron rods 3 to 4 feet or more in length, of  $\frac{1}{4}$  to  $\frac{1}{2}$  inch thickness; at one end formed to a close, flat spiral, making a disc from 2 to 4 inches diameter at an obtuse angle to the rod, which is suitably bent for convenient use in removing slag from the surface of molten metal.

**GLOVES.**—Made of canvas, with a thumb, but not separate fingers; padded on the back. Used to protect the hands of the melter from the heat. A narrow bag of doubled sackcloth, or blanket covered with canvas, into which the arm can be thrust to the shoulder, is as good, and much cheaper.



**BAR MOULDS.**—Made of cast iron. Used to pour the molten metal into in order to form a bar or ingot.

Ounces, silver,	15	27	52	107
Ounces, gold,	28	50	107	200
Price, each,	\$0.35	.50	.75	1.25
Ounces, silver,	160	265	428	683
Ounces, gold,	300	495	800	1,000
Price, each,	\$2.25	3.20	3.50	4.50

Larger sizes can be had up to 2,000 ounces of silver.

**STIRRERS.**—Strips of the same material as the crucibles. Pieces sawed from the sides of worn-out pots answer. Used for stirring the molten metal.

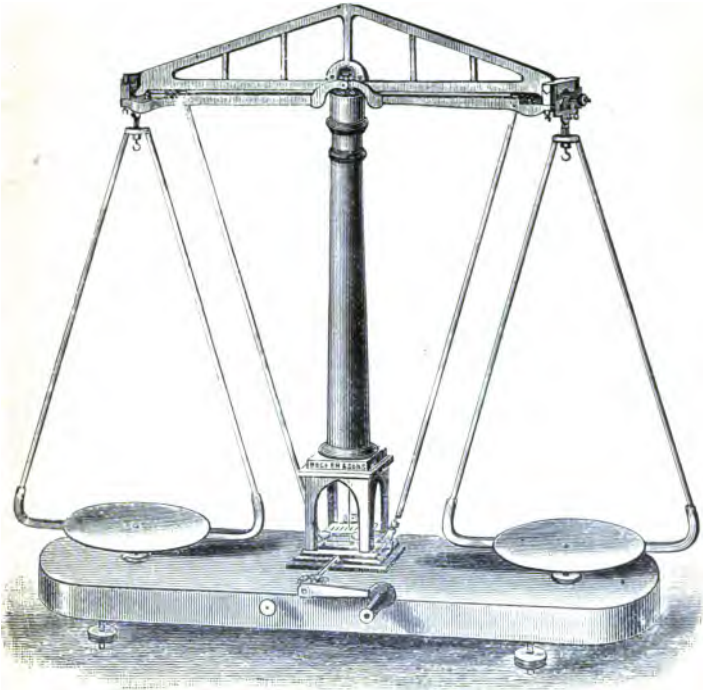
**HAMMERS, CHISELS, ETC.**—Round-faced hammers for smoothing bars, and chipping hammers for driving the chisel, of different sizes, from 4 ounces to 4 pounds, according to the bars. Thin sharp cold-chisels for chipping. Cutting pliers, also scissors, and a small file for adjusting the weight of bullion assays.

**SUNDRIES.**—A strong table should stand near the melting furnace on which to place the crude bullion, as gold-dust, or lumps of retorted amalgam, etc., in sheet-iron pans, in readiness for melting; also to place the cleaned bars on, etc. There should be a rack in which tongs and fire-irons may rest standing on end on the bricked floor, conveniently for use; a simple rail with some nails projecting will serve. Some put the nails in the edge of the table, which then stands behind the melter, who has only to turn around and put his hand on what he requires. Also a tub of water in which to cool the bars, with a sloping platform adjoining, on which to clean them, arranged to drain into the tub; a box of sharp sand for scouring the bars, a scrubbing brush, and a tight roll of canvas about  $1\frac{1}{2}$  inches thick,

the wetted end to be dipped in the sand and applied to the bars as a scourer. For polishing gold bars some use a bundle of brass wires as a brush. Diluted sulphuric acid, in a tub or vat, is sometimes used for "pickling" a bar containing base metal to improve its appearance, and a bottle of acid is usually kept at hand for use in the scouring. A block of timber set on end, the top covered by sheet zinc and inclosed by a rim or ledge, is required to place bars on for chipping; some small sheet-iron pans for samples (*chips or dips*), and larger ones for crude metal.

STAMPS.—Letters, figures, dollar sign, No., Oz., and words, such as Fine, Value, Total, Gold, Silver, and the name of the assayer; made of steel and used for marking the bars. Sizes, from  $\frac{1}{8}$  inch to  $\frac{3}{4}$  inch. Should be arranged in holes in a block of wood for convenience.

BULLION SCALES.—These scales, though strong and massive, are also delicate, and are provided with apparatus by which the respective bearings are relieved when not in use; also with an arrangement by which the pans are steadied while the weights are adjusted. In using the balance, the object to be weighed and the heavier of the necessary weights, must be placed on the pans before the beam is suspended on its bearings by a turn of the crank in front of the base. The lighter weights for completing the weighing are then



BULLION BALANCES AND WEIGHTS.

adjusted while the pans are steadied by the arrangement spoken of above, and released by a touch when desired. Scoops and pans for gold-dust, etc., accompany the scales.

In large establishments two pairs of bullion scales are used, one very massive pair for large quantities of metal (chiefly silver), and a lighter pair for smaller lots, especially gold. The larger balance has only the

upper part inclosed in a glazed case; the gold scales are completely inclosed. In silver mills, platform scales are often used for the weighing of the bars; they can be obtained to show both troy and avoirdupois weight, or the latter may be converted into troy by calculation, or by means of the assay table for 20 grammes of ore in Part I.

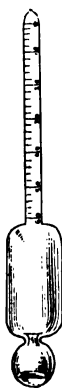
**FLASKS.**—Thin glass flasks, used for parting bullion



assays, and for other purposes. Three-ounce, flat-bottomed matrasses are suitable for bullion parting.

Flat Bottom, per doz.,	\$1.10	\$1.25	\$1.50	\$1.80	\$2.25	\$2.60
Capacity in oz.,	$\frac{1}{2}$	1	2	4	6	8

**HYDROMETER.**—A floating gauge, made of glass and loaded with shot at the lower end. Placed in a liquid

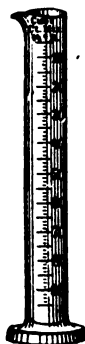


it floats in a vertical position, a greater or less portion of the graduated stem being submerged according to the density of the liquid; used as a measure of the density, and therefore the strength, of acids, etc. There are several kinds of hydrometers; that of Baumé is generally used in assay offices. In ordering a hydrometer, state the purpose for which it is required, because some are arranged for liquids lighter than water; these would not answer for acids. It is best to procure one which is graduated to show specific gravity as well as degrees

Baumé, because the strength of the acid is indicated in either in the books. Price, 50 cents.

**CHARRED LENTILS.**—Made by heating lentils in a closely-covered vessel until no fumes are given off. Boiling acid is liable to *bumping*; this is prevented by placing a small piece of charcoal in the matrass.

Common charcoal answers, but charred lentils throw off no fragments.



**GRADUATED CYLINDER.**—The hydrometer is used in a narrow glass vessel, which need not be graduated for this purpose, but a graduated vessel is useful, and it answers equally for floating the hydrometer. As to the graduation, grammes and c. c. are the same, a gramme being the weight of a c. c. of pure water at standard temperature.

Cylindrical, with lip, double graduations in cubic centimeters, reading either up or down.

Capacity.....	5 c. c.	10 c. c.	25 c. c.	50 c. c.	100 c. c.	200 c. c.
Each.....	\$0.30	.35	.50	.65	.85	1.00



**BEAKERS.**—Thin glass jars, which will bear rather rapid change of temperature without fracture. Used



in treating substances with acids, etc., and for receiving the liquid from a filter (*filtrate*). Not essential in the ordinary routine of assaying gold and silver, but very appropriate in an office, and requisite in the assays of copper and other base metals.

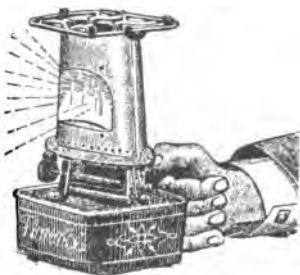
Made of Bohemian glass, lipped or plain, equally thin at bottom and sides; in nests.

Nos. 1 to 3,	capacity 3 to 7 oz.,	per nest \$ .45,	3 in nest.
Nos. 1 to 5,	" 3 to 16 "	" " 1.00,	5 " "
Nos. 1 to 7,	" 3 to 36 "	" " 1.75,	7 " "
Nos. 1 to 10,	" 3 to 90 "	" " 3.50,	10 " "



EVAPORATING DISHES.—Made of glazed porcelain. Will withstand heat and acids; used for dissolving and drying; not essential in assaying gold and silver.

Nos.....	000	00	0	1	2	3	4	6	8
Capacity, oz....	2	3	4	6	8	16	32	64	96
Each.....	\$0.20	.25	.30	.40	.50	.65	.75	1.25	1.75



COAL-OIL STOVE.—Useful anywhere. Almost indispensable in a country office where gas cannot be had. A sheet-iron dish containing some sand, placed on the stove, forms a sand-bath convenient for the parting.

One Burner, iron system .....\$0.75

FLATTING MILL (Rolling Mill). A pair of steel roll-

ers with adjusting screws and cranks to operate by hand. Used in laminating metal; can be dispensed with, but not conveniently, by using the hammer and anvil instead. Prices from \$30 to \$95.\*

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\* The apparatus mentioned and illustrated in the course of description of methods can be had from John Taylor & Co., San Francisco, Cal.

## MELTING BULLION.

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THE fuel used is coke or charcoal, or in some places stone coal; it should be screened free from dust and dirt. Lumps larger than a turkey's egg should be broken. When coke is used, a little charcoal is necessary to start with.

New black-lead pots must be annealed. To anneal a pot and cover, kindle a fire in the furnace and invert the pot over the fire; put the cover also in the furnace; add fuel, check the draft, and increase the heat very gradually until the pot and cover are red-hot; they are then ready for use, and may thereafter be placed, even when cold, in the hottest fire without danger of fracture.

The metal to be melted, if in small quantity, is put into the cold (annealed) pot as it stands on the table, or on the furnace plate; the pot is then placed in the fire, resting on a piece of fire-brick. Otherwise the pot is put into the fire first, and large bars or lumps are placed within it by means of tongs, gold-dust or small fragments of metal by means of a scoop and sheet-iron funnel. Borax is added, the pot covered, and fuel placed around and above it. Retorted amalgam, especially silver, is porous and bulky; hence the pot must be refilled from time to time as the metal

subsides, and as such metal contains some quicksilver, the addition should be made before that in the pot is quite melted, while it is in a pasty condition, otherwise spirting may occur. The melter should be careful not to inhale the fumes.

When the metal, and the slag formed by the borax with the impurities, are melted, the latter must be removed. The skimmer is placed in contact with the slag, of which a portion adheres; the skimmer is then withdrawn, pressed on the furnace plate previously greased, or on any suitable surface, to chill and flatten the adhering slag, dipped slightly in cold water to further cool it, and again touched to the slag. If the slag is in such quantity as to accumulate inconveniently on the skimmer, it is thoroughly cooled in water and knocked off by a blow with the hammer; toward the end, however, it is better if the skimmer is not cleaned, because the metal is less liable to adhere to a surface of slag than to the naked iron.

The slag must be of medium consistency, neither pasty nor too liquid; if pasty, it may retain considerable globules of metal; if too thin, it cannot be readily skimmed off. A thick slag may be thinned by an addition of borax; or, if sand be the cause, a little soda may be employed, but as soda attacks the pot it must be used with care. A thin slag may be thickened by means of sand.

When all slag has been removed, if the surface of the metal is bright, it is ready for pouring. If it is covered by a scum or crust, it probably contains copper or iron, perhaps also sulphur. If rainbow-colored

rings are seen, lead is most likely present in small quantity, and more if brilliant spots appear moving rapidly.

In the first case, some lumps of borax are to be thrown on the metal and allowed to melt, absorbing the base oxides, and forming a slag which must be removed as before; if this does not seem to be efficacious, sulphur is probably present, and a little nitre may be added to the borax, or, better, a mixture of nitre and glass powder. If much copper or iron is present, it is best to add a little lead to the metal in the pot; the colored rings or bright spots then appear, being produced by the formation of litharge, which rapidly oxidizes the copper and iron, forming slag. As the plumbago of the pot is a reducer it constantly tends to the reconversion of the litharge to lead, and of other oxides to the respective metals, while litharge being a flux for all earthy matters, attacks also the clay of the pot. To prevent this reverse action and protect the pot, some cupels are introduced which float on the liquid metal and absorb the litharge, etc. The cupels are removed when saturated. Unless the bullion is very nearly all precious metal, the refining by these methods is too tedious, but sulphur should be removed in order to make a bar which can be correctly assayed.

While the melting is in progress, a stirrer is heated to redness, and a suitably-sized mould is warmed. To prevent adherence of the metal, a mould for a gold bar is smoked by being placed in a small chamber, or mounted on two brickbats, over a dish of burning

resin, or a quite small mould, by holding it over the flame of a lamp; it is thus warmed at the same time. For a silver bar of any considerable size, greasing is usually preferred; or the mould may be painted with a wash of finely-sifted ashes and water, then thoroughly dried and warmed. Paraffine, the mould being warm enough to melt it, makes the best greasing.

After the skimming, the metal in the pot is covered by a layer of charcoal powder and left for a few minutes, the pot being covered; this reduces any base metal oxide that may have formed, and which would cause the metal to sputter when poured, or it prevents the sprouting of nearly pure silver. A silver, doré, or base silver bar, if of considerable size, must now be sampled (all very small bars are chipped). The pot is taken from the fire, placed on the furnace top, and the metal stirred by an assistant, with the heated stirrer held in the tongs, while the melter dips out two separate samples of 30 to 50 grains each, and pours them, from a height of a foot or two, into dishes of water, by which the metal is granulated. The dipping is usually done with a very small crucible, heated to redness and held in a pair of tongs. A heated rod of iron is often used for the stirring, and, if the end is formed into a small ladle, for the dipping also; an assistant is not then required. The metal is then poured into the bar-mould, the charcoal being held back, if necessary, by means of an iron, such as the bent poker, until nearly all the metal has run out. Some more charcoal dust is then thrown on the bar to keep the surface covered, or, if the bar is small,

some oil may be poured on it; the purpose is to prevent oxidation. The slight cooling of the metal during the stirring and sampling does no harm, for if silver is poured too hot, the face of the bar will be spongy. The granulated samples, or "dips," are dried, and are then ready for the assayer. A gold bar is poured as soon as melted and stirred, the samples not being taken by dipping.\*

In some melting rooms the heaviest bars are cast without the use of a crane or of basket tongs. Two men grasp the pot on opposite sides by means of straight "side tongs," the jaws of which are formed to suit the thickness of the pot, so as not to bite a piece out, and it is lifted out of the furnace in less time than would be required to adjust the basket tongs. The pot is tilted for the pouring by means of a pair of tongs, which are held in a horizontal position, having semicircular jaws grasping the pot about the middle. This way of pouring requires considerable strength, and the basket tongs, suspended from a crane by means of an eye formed on the pivot of the tongs, and having the handles secured by a ring slipped over them, seem preferable for heavy work. At Los Bronces, in Mexico, the furnace was set in the ground, the ash pit covered by an iron grating on which the workmen stood. The pot contained enough metal for a number of bars; it was lifted by a crane, and placed in a carrier such as is used by foundrymen for carrying melted iron to the flasks, and two men lifted it and filled the moulds in succession.

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\* As a guide in selecting a mould of suitable size, it is well to remember that a cubic inch of gold is very nearly 10 ounces.

When the bar is solid, it is turned out of the mould by upsetting that, cooled in water, any slag beaten off, scoured, smoothed if necessary by hammering, and dried; a bar which has been sampled by dipping is now ready for weighing, otherwise it must first be chipped. The chips are taken from two diagonally opposite corners, one top and one bottom. The chisel is held between the thumb and forefinger of the left hand, while the remaining fingers of that hand retain the chips, which would otherwise be projected to a distance. The chips are taken in many small pieces, to facilitate the weighing of the assay, and each sample is placed on a separate piece of paper, one corner of which is bent up for convenient handling. The number of the deposit is marked on the paper. One assay is made from each sample, and if the two do not agree, and a "control" assay fails to correct the discrepancy, the bar is remelted and better mixed. The samples should be large enough to allow of several assays being made of each, and any residue is folded in the paper, the number being left visible, and kept for future reference in case of need. The chipped corners of the bar are neatly dressed by the hammer, and impressed with a stamp as a security against being tampered with. The bar is then weighed, and a tag bearing the number and weight pasted on, to remain until the stamping; or the number and weight may be stamped at once.

Each bar is finally stamped on the face, which is that side which was the bottom when cast, with the name and address of the assayer, the weight in troy



ounces and hundredths, and the fineness and value in gold, or in both gold and silver, according to the character of the bullion, as explained above. On delivery it is accompanied by a certificate, stating the number of the deposit, the name of the depositor, the weight before and after melting, and all other particulars as stamped; also the charges for melting and assaying, against which is offset the value of the *chips* or *dips* and *grains*. The dips or chips are the samples; the grains are any globules of metal which may remain in the pot after pouring, or which may be removed with the slag in the skimming, all of which must be collected, cleaned, and weighed. A convenient way in which to arrive at the weight of the chips and grains is to weigh the bar and grains together before chipping, then chip and weigh the bar alone for its delivery weight, the difference is the weight of chips and grains, the value of which is reckoned according to the result of the assay. The value stamped on a bar is the assay value, the commercial value depends on the state of the market for the particular description of bullion.

Gold is frequently purified from base metal in the following manner: The melted metal is slowly poured from a height of several feet into a vessel containing water which is meanwhile agitated by means of a stick. The metal is thus granulated; it is then dried and remelted with addition of nitre and borax. The melting with nitre must not be done in a black-lead pot, but in a "sand pot," or one of clay. The nitre in decomposing leaves caustic potassa, which violently

attacks the pot, as do also the base metal oxides formed; this may lead to fracture, when the spilled metal must be recovered, as far as possible, by washing the ashes and cinders after allowing the fire to burn out. To prevent such an accident two devices are employed: Firstly, a dish, made by cutting down a worn-out black-lead pot, is placed under the sand pot in the furnace, as a saucer under a cup; this receives the metal in case of a break; secondly, a quantity of clean quartz sand, equal to about half the bulk of the nitre, is mixed with that and the granulated metal. The sand combines with the metal oxides and the potassa, forming a liquid slag or glass. Without this addition of sand, iron or copper oxide would not become liquid unless litharge were also present or a large proportion of borax were used. The metal and slag are poured together into a mould. Sometimes a very neat bar is thus made, which does not require remelting, but frequently the slag gets between the metal and the mould, spoiling the appearance of the bar, and making it difficult to clean; in this case the bar is remelted with a little borax in a black-lead pot. If, however, the metal is not pure enough, the granulation and melting with nitre are repeated. Retorted amalgam does not require granulation being porous enough for the melting nitre to permeate it.

The fusion with nitre must be done carefully and under a very low heat for some time; otherwise the contents of the pot will overflow. The pot should not be more than half filled with the mixture. It is not safe to attempt a second use of the sand pot, as it is almost certain to break.

A very good way in which to purify small lots of gold or silver containing base metal is by cupellation. A worn-out black-lead pot is cut down so as to form a dish from 2 to 4 inches deep, in which a cupel is made by beating in moistened bone ashes, and scooping a hollow in the top. The cupel is well dried in the dish, which may have some holes drilled through it with advantage. When required for use, the cupel is placed in the flue of the melting furnace, while the fire is in use for other purposes; as soon as the cupel is red hot, some lead is placed on it, and when that is melted the impure gold or silver is added and cupelled without expenditure of time or much fuel, because the melting of other lots may be going on in the furnace. The furnace must be left a little open to admit air for the cupellation.

A few ounces of metal may be cupelled in the ordinary muffle, a cupel being made in the following manner: A strip of cotton cloth is laid across a small bar mould, and pressed down into it, the ends of the cloth projecting; moist bone ashes are then beaten in until the mould is full; a depression is formed in the top by means of a knife, and smoothed by the bowl of a spoon. The cupel is then lifted out of the mould by means of the strip of cloth which passes under it, and dried; or a cupel may be made in a ring of hoop iron or tin, and used with the ring still on it.

Refining as described does not remove silver from gold bullion. In Australia, silver and copper are removed from gold by injecting chlorine through a clay tube (pipe stem) plunged into the melted metal;

the chlorine combines with the copper and silver; the copper chloride volatilizes; the silver chloride floats on the gold and is poured off as soon as the gold solidifies by cooling, after which the gold is again heated to melting and cast in a mould.

## ASSAYING BULLION.

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IN bullion assaying the object is to find out how many parts in one thousand equal parts by weight of the bullion consist of pure gold or silver; or, which amounts to the same, mathematically, how many .001 of 1 part are gold or silver. In writing, and in stamping the bar, the latter view is taken, and we use the decimal mark thus: fine gold, .572; fine silver, .422; that is, so many thousandths of a part in one part of the alloy. But in speaking we say, the bullion is so many fine in gold or silver, not mentioning but implying thousandths. Moreover, in speaking, the thousandths are often called "points," as, "this bar is four points finer than that," or, "add three points for cupellation loss."

For gold bars, using a delicate balance, half a gramme is generally taken for the assay, the purified metal being weighed in half milligrammes. With an inferior balance, or for a silver bar, one gramme or ten grains are the usual quantities; whichever it be, the weight taken is called a thousand, which is understood to mean, a thousand thousandths. Hereafter in these pages we shall speak of thousands and points, discarding the awkward word thousandth.

In bullion assaying, all the weighing, except that of the lead used, is done on the assay balance.

**GOLD BAR.**—Natural gold is never pure; it is alloyed almost invariably with silver, frequently with copper, and the bars often contain platinum and iridium, which occur in grains with the placer gold, lead, from bullets scattered about the gravel washings, and quartz-mill gold sometimes contains iron from the abrasion of stamps. All base metal can be removed by cupellation with lead; silver and platinum, by nitric acid; for, although pure platinum is not attacked by nitric acid, it dissolves in that when alloyed with two parts of silver; and as gold must also be alloyed with at least twice its weight of silver, in order that the parting may be complete, it will be readily seen that the presence of platinum offers no serious difficulty. Iridium does not melt to an alloy with the gold, but remains in the form of scales or grains.

It will be remembered by those who have studied Part I that, while two parts of silver to one of gold are indispensable to a successful parting, more than three parts would cause inconvenience by the crumbling of the gold; moreover, it has been ascertained that with more than three parts the separation is less perfect than with two and a half parts, until the proportion of eight parts of silver to one of gold is reached, when the gold falls to absolute powder and remains pure.

The assayer endeavors to obtain an alloy for the parting in which the proportion of silver to that of gold shall be about two and a half to one, supposing platinum to be absent. A practised assayer can usually judge from the appearance of the sample, or from former experience with gold from the same local-

ity, how great a proportion of silver it already contains, and consequently, how much must be added; otherwise a preliminary assay may be made with addition of so much silver as to assure the success of the parting, even though the gold may crumble, and thus the requisite information may be obtained.

**PRELIMINARY ASSAY OF GOLD BAR.**—One thousand of the sample and four thousand of test silver are enveloped in three or four thousand of sheet lead, and cupelled under a rather high heat. The bead is cleaned, beaten by hammer and anvil to a flat plate, annealed by heating to redness, parted, cupped, dried, and weighed, as in ore assaying, but a matrass is used for the parting, instead of a test-tube, on account of the larger quantity of metal. The weight in points is the approximate fineness from which the weight of silver required in the proper assay is known by multiplying by 2.5.\* From this the quantity already alloyed

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\* In the case of very small bars, this assay will suffice, if the gold be twice boiled in acid, well washed, cupped with care, and at least two points deducted from the result found. It is not absolutely necessary to take exactly 1,000 of the metal for an assay. Where bullion assaying is a regular business, it is best to follow the regular system, and avoid calculation as much as possible; an operator soon learns to weigh out the exact quantity required in an expeditious manner; but where a bullion assay is only an occasional incident, it may be found more convenient to take such a weight as may be readily got without paring and filing. For a preliminary assay, with a fine balance, any weight between 100 and 1,000 will suffice, and this being inquartated and parted, the approximate fineness may be calculated by proportion.

Suppose we take a piece, or two pieces, weighing 236 points, and we get 233.2 points of fine gold; we have only to add ciphers to the latter number and divide by the former to three places of figures in the quotient, giving .998 as the fineness.

with the gold must be deducted, in order to ascertain how much must be added. For example, if the gold got from 1,000 of the sample weighs 924, the lost weight is that of the silver, except, perhaps, a very small quantity of base metal, which need not be noticed in this class of bullion; the bullion, then, contains 924 of gold and 76 of silver in 1,000. Multiplying 924 by 2.5, we have 2,310 of silver required for the parting, of which quantity the bullion already contains 76, leaving 2,234 to be added.

**ASSAY PROPER OF GOLD BAR.**—One thousand of the sample is cupelled with such a quantity of test silver as is necessary, about 3 points of copper, and about 3,000 of sheet lead, rolled into a capsule or "cornet" and closely folded about the assay. The lead cornets are usually kept at hand, ready made. The weighed assay is transferred directly into the cornet, held in the left hand, from the weighing capsule which is lifted from the scale pan with the pincettes. It is not usual to weigh the silver for the inquartation; a quantity of sheet silver is cut into pieces of nearly known weight, and the required number of pieces, as well as the copper, added to the gold in the lead cornet, which is folded in the form of a snug packet and placed on a watch-glass, or in a dry cup, which occupies its appropriate place on a tray according to the set number of the assay.

As many as 14 assays may be thus prepared and cupelled at one time in a mint muffle. The cupels are heated to light redness, and the assays are placed on them in regular order, according to their respective



numbers in the set; the muffle is then closed until the assays *uncover*, when the door is left open. If there is any difficulty in keeping the front cupels hot enough, some glowing coals may be placed in the mouth of the muffle. It sometimes happens, in cupelling a number of assays, that the uncovering of one or more is delayed by some cause; a small wad of folded paper, placed on the refractory assay, will set it working. A high heat is maintained throughout the operation, and especially at the time of brightening, that is to say, a higher degree than is admissible in a silver assay, because gold is less volatile than silver, as well as less fusible.

It is not necessary to remove a gold assay the instant the cupellation is finished, so that all may remain in the muffle until all are finished, and may then be taken out without much precaution as to cooling, as these assays are not liable to sprouting; the addition of copper is partly for the purpose of preventing the sprouting, for, notwithstanding the cupellation, a trace of it remains in the bead. Another reason for the addition of copper is that it renders the metal tougher for the lamination. If, for want of heat, an assay should *freeze*, or solidify, during the cupellation, it may be remelted by placing glowing coals about the cupel, and one upon the top of it, but the assay is not quite reliable.

The cupelled bead is cleaned, beaten to the form of a bar about one-quarter inch wide and one-eighth thick, annealed by heating to dull redness in the muffle, and passed through the flattening mill until it is reduced to

a strip about as thin as an ordinary card. The metal must be annealed whenever it shows signs of cracking on the edges, or becomes hard and elastic from the rolling, and again after the rolling is finished; it is then formed into an open coil\* by the thumb and finger, placed in a three-ounce matrass and *gently* boiled in one and a half ounces of nitric acid of 32° Baumé, adding a charred lentil or a bit of charcoal to prevent bumping. After thus boiling for 10 minutes, the acid is poured off, replaced by a fresh quantity, and the boiling repeated for 15 or 20 minutes. It is in the second boiling that the charcoal is especially necessary, as there is no danger of the bumping of the acid until the evolution of gas nearly ceases.

The acid is now again poured off, and this time the charcoal must go with it, or, on the addition of water it will sink and give trouble. The gold is washed three times with hot water, cupped, heated to redness in the muffle, cooled, and weighed; it should be in one piece, which, dark colored and fragile after the parting, assumes the golden hue and a fair degree of tenacity when heated. The color, although golden, is liable to considerable variations of shade.

The weight of the gold in points, less from half a point to one or two points allowed for a small portion of silver, which remains in it and is called the *surcharge*,† is the gold fineness of the bullion, which is

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\* The name of "cornet" is given to this as well as to the lead capsule. In case you have not a flattening mill, the hammer and anvil must be made to serve in its place.

† A larger surcharge is allowed for very small bars than for large ones; also when a little base metal is believed to be present, the bar being brittle, etc.

stamped on the bar. The results of two assays should agree within a quarter point in weight.

**DORÉ BAR.**—The assay is conducted, speaking generally, in a similar manner to that of a gold bar. A preliminary is more often necessary in this case, and according to its indications either gold or silver may be added so as to produce a suitable alloy for the parting; if gold is required, it is taken from the cornets of previous assays, or pure gold may be bought for the purpose.

In the assay proper, if gold is required it is weighed, added at once to the weighed assay, and the whole cupelled with a suitable quantity of sheet lead. The difference between the weight of the bead and that of the assay *plus the added gold* represents base metal. The bead being laminated, parted, etc., the further loss of weight is the silver fineness to which a correction may be added, according to the judgment and experience of the workman, for cupellation loss, from one to three or more points. Finally, the weight of the cornet, less that of the added gold and a suitable allowance for surcharge, is the gold fineness. Except in particular cases, the inquartation by addition of gold is generally omitted.

If an addition of silver is necessary, as, for instance, in the case of a bar which is about half gold, it, like the gold, may be added at once, and allowed for in the final estimation of the silver fineness. It is, perhaps, better to cupel the assay with the requisite quantity of lead to clear it of base metal, note the weight of the bead, then add the silver and cupel with a very light

lead cornet, laminate, part, etc., and weigh the gold; the difference between the gold fineness and the weight of the bead after the first cupellation, plus a suitable correction for loss, is the silver fineness. By operating in this way, the cupellation loss of the added silver need not be considered.

Example: A thousand of doré bullion containing some copper was cupelled with 12,000 of lead, and gave a bead weighing 900. It being supposed to contain too much gold for parting, 2,000 of silver were added, and the whole wrapped in lead and again cupelled, then parted; the gold weighed 400. Deducting 400 from 900 left 500 for the approximate weight of the silver. In the assay proper  $400 \times 2.5 = 1,000$  of silver, was requisite, the bullion contained 500, hence 500 of silver must be added.

One thousand of the bullion was then cupelled with 8,000 of lead, and gave a bead weighing 907, which was again cupelled with 500 of silver and a small lead cornet, then laminated, parted, etc.; the gold was in a single piece and weighed 401. The bar was therefore 401 fine gold, and  $907 - 401 = 506$  fine silver, to which a point might be added for cupellation loss. Unless in particular cases, or for quite large bars, the process indicated for the preliminary, if executed with skill and judgment, answers all purposes.

BASE BAR.—This assay is better scorified before cupellation. An approximation to the quantity of lead required, and to the gold and silver fineness, may first be obtained by cupelling 1,000 of the bullion with as much lead as may be found necessary for clearing the

bead. Beginning with the quantity of lead which is judged to be required, and watching the operation, more lead can be added from time to time until the bead is seen to round up, and to exhibit the brilliant play of colors immediately preceding the brightening. Silver is to be added, if supposed to be requisite for the parting; it must be weighed. In the assay proper, 1,000 of the bullion may be scorified with the proportion of lead found necessary in the preliminary, and a little borax. When the scorification has been carried far enough, the assay may be poured, or cooled in the scorifier and that broken, the button placed on a hot cupel, and any desired addition of gold or silver made. The gold or silver must be weighed, wrapped in sheet lead, and added to the button after that is melted; or the addition of silver may be deferred until the bead is cleared, cleaned and weighed, as in the assay of a doré bar, and for the same reason, yet if the bullion contains much copper, and but little silver in proportion to the gold, it is better not to postpone the addition of silver, because in the cupellation of gold and copper, without a considerable proportion of silver, some gold is lost. For this reason, on the other hand, the addition of gold when much copper is present is better postponed until the copper has been removed. Neither gold nor silver should be added during the scorification, as needless losses are incurred thereby, all of which will inure to the detriment of the bullion.

It has been observed by experimenters that if gold is added to lead already melted on a cupel, little or no loss occurs, but if the gold be previously scorified with

lead, and then cupelled, there is a notable loss. As with doré bullion, the inquartation by addition of gold is not generally practised. Where so much lead is used as sometimes in the assay of base bullion, it need not be all sheet lead; pieces of assay bar lead may be used on the cupel, and either that or granulated lead in the scorifier, so that sheet is only required for wrapping the assay and the gold or silver for the inquartation for which it is safer than the paper recommended by some writers, though paper may be used in case of need.

Example of assay of base bar containing lead and copper:—

Preliminary cupellation without addition,

gave a coppery bead, weighing..... 732

showing that the assay contained about..... 268 lead

recupelled with 10 parts = 7,320 lead

did not clear; added 2 parts = 1,464 lead

which cleared the bead. Total lead.....8,784

The bead weighed.....327

Added an equal weight of silver; cupelled and

parted, gold weighed..... 47

Silver.....280

In the assay proper, 1,000 scorified with 8,500 of lead  
and cupelled with addition of  $\frac{280}{25} - 47 =$

65 gold, weighed .....	396
Parted; gold weighed .....	112½
Silver weighed .....	283½

True weight of gold-(after deduction of 65 added  
from the total)..... 47½

The assay therefore shows the bar to contain in 1	
part, fine gold .....	0.0475
“ silver .....	0.2835
Base metal .....	0.6690

1.0000

If the proportion of gold to silver had been reversed, the bead from the preliminary would have been yellow; we should then have added twice its weight of silver in the preliminary and 2.5 times 280 of silver in the assay proper.

The following table, from Mitchell, will give an idea of the proportion of lead required to separate copper from gold by cupellation:—

GOLD IN ALLOY.	LEAD REQUIRED.
.900 .....	10 parts
.800 .....	16 “
.700 .....	25 “
.600 .....	24 “
.500 } to .050 }	34 “

For alloys of copper and silver, about half the above proportions of lead suffice, but in both cases much depends on the temperature of the muffle and on other conditions; the guide is the condition of the bead; if it is hard, brittle, or coppery, it must be treated with a fresh quantity of lead.

**GOLD AND PLATINUM.**—The presence of platinum in the assay is known by the following signs: The brightening of the bead requires a much higher heat than usual, and is less vivid. The bead is flat, thick-edged, and dull. On parting, if the platinum is in the proportion of 1 or more to 50 of gold, the acid is colored straw yellow. The annealed cornet is paler than usual, even to resembling tarnished silver. It is important to recognize the presence of platinum in gold bullion, lest it be mistaken for silver, because, while not removed by cupellation, it is dissolved in the parting. In order that the parting may be effected in the presence of platinum, the quantity of silver alloyed must be as much as twice the weight of the gold and platinum together, and as both the silver and the platinum are dissolved in the ordinary parting with nitric acid, it may happen that the gold will crumble.

An alloy of gold and platinum with 12 parts of silver may be parted by boiling in sulphuric acid, leaving the gold and platinum, the silver alone being dissolved. The gold and platinum are washed with hot water, dried and weighed, then again alloyed with  $2\frac{1}{2}$  parts of silver and parted with nitric acid, leaving the gold, which is washed, dried and weighed. The



more usual method is to make an alloy with enough silver to part in nitric acid, which leaves the gold; then to precipitate the silver from the liquid, in the form of chloride, by adding hydrochloric acid. The silver chloride is separated from the liquid by filtration or by decantation (pouring off), dried and weighed; 75.27 per cent of the weight is silver,\* from which the weight of the added silver must be deducted, leaving the silver fineness of the bullion. To the liquid containing the platinum is added a little hydrochloric acid, and then solution of ammonium chloride as long as a precipitate forms. The liquid is then evaporated to dryness, and to the residue, when cold, dilute alcohol is added, and the insoluble portion (platinum ammonium chloride) collected on a filter, washed with alcohol, dried and heated to redness; the residue is platinum, which is weighed. The united weight of the gold and platinum, deducted from that of the cupelled bead (minus any added silver) leaves the silver fineness of the bullion, which should agree nearly with that found by calculation from the precipitated chloride as above.

Iridium does not form an alloy with the gold; it mostly falls out of the cornet during the lamination, in the form of scales of a steely color; if in large quantity, a part may be found at the bottom of the pot in which the bullion is melted.

**SILVER BAR.**—Silver bullion usually contains copper or lead, or both; the proportion of lead required to

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\* If the drying has been finished at the temperature of incipient fusion.

remove the copper depends on that of the copper to the silver. If the quality of the bullion is not approximately known, it may be ascertained by a preliminary cupellation with as much lead as may be found necessary, the assay proper is then treated accordingly. Any lead which the bullion may contain is counted as a part of that required, hence if the bullion contains some lead, 1,000 of it may be cupelled without other addition than the small piece of sheet lead necessary to envelop it. If the bead clears, no further addition is required; if not, lead is added in successive small portions until it does, and the quantity of lead so added is approximately that which will be requisite in the assay proper. A table will be given which will aid in the apportionment.

If much lead is required, it is best to melt the greater portion of it on the cupel before adding the assay; bar lead should be employed in this case. When the lead is quite melted, the assay is added, enveloped as usual in a lead capsule, the weight of which makes up the required quantity. In order to make a proper correction for the cupellation loss, a *test assay* is made at the same time and in the same heat. Test silver and copper are weighed off in the proportions in which they exist in the bullion, as nearly as may be judged, or as indicated by the preliminary if made, and cupelled with the same weight of lead as is used for the assay; the loss sustained by the test, or for safety a point less, is added to the result of the assay. Unless the bar is quite small, two assays, one from each of the two samples, should be made, and should agree within a point.

The cupellation must be conducted with great care as to heat; I have known as much as 30 points to be lost by unskillful cupellation, and, although a test or check assay may be made with each bullion assay, or in the midst of a group of those of approximately similar quality, yet, when losses are excessive, the check cannot be implicitly relied on for the correction. Tables of the loss for bullion of various grades of fineness are given in books, but are only useful as showing what has been, and therefore can be, done. The loss should seldom be so much as 10 points, and with bullion that is nearly fine should not exceed 3.

The smoke from the cupels should rise to about the middle of the muffle, the draught through that not being so strong as to draw it immediately through the holes, a part generally escaping by the front. If much lead is used, crystals of litharge should form on the cupel at a little distance from the bead, and even the brightening heat should not cause them to disappear; this is called "feathering" the cupel. When the bullion is so fine as to require but little lead, it is scarcely possible to feather the cupel without great risk of freezing the assay, and an assay once frozen, even though remelted, is not reliable.

On account of the delicacy of this operation and the occasional necessity for moving a cupel during its progress, as well as at the finishing, only a small number of assays can be properly made at once in a muffle which is only 4 or 5 inches wide. When the bead is about to brighten, the cupel containing it should be pushed further back where the heat is higher,

or a glowing coal may be placed near; it must remain in this higher heat only a few seconds after the brightening, say while five can be deliberately counted; it must then be cooled with the precautions indicated in Part I, the best portion of which, being a method not generally published, may be repeated here.

The cupel is drawn to the front of the muffle, and gently tapped on one side by the tongs. At the instant when the bead ceases responding to the taps by its vibration, and a certain change, which will be recognized when once seen, comes over it and the face of the cupel, indications of the solidification of the bead, it is pushed back into the hottest part of the muffle, and there left for a minute or thereabout, after which it will not sprout, being solidified all through, as shown by a dimple on its surface caused by contraction.\* The heat must not be so intense as to remelt the bead, or the effect desired will be lost, as will also some silver; but a bead is not very easily remelted when once chilled. The bead must be white, well rounded, *slightly* adherent to the cupel, and crystalline at the bottom when cleaned; it is weighed, and then parted without addition of gold to inquartate. Any gold found is cupped, dried, weighed, giving the gold fineness, which, deducted from the weight of the bead, leaves the silver fineness, to which is added a correction indicated by the check. The following table from Mitchell gives the approximate proportion of lead

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\* The most accurate results are got when the bead solidifies, at the instant of clearing, but it is not easy to hit the right point as to temperature so as to remove all the lead.

required for the cupellation of different alloys of silver with copper:—

SILVER IN ALLOY.	LEAD REQUIRED.
1.000 (pure silver).....	.300
.950.....	3.000
.900.....	7.000
.800.....	10.000
.700.....	12.000
.600.....	14.000
.500 to pure copper.....	17.000

The reason that 1,000 of pure silver requires 300 of lead is that silver alone cannot be melted on the cupel without its throwing off minute globules, nor can a good bead be formed without the aid of lead; so that no matter how nearly pure the silver may be, that quantity of lead, at least, must be used.

**SILVER LEAD.**—This assay is very simple; it is only necessary to wrap the weighed assay in a lead capsule, in order to insure the union of all fragments, and cupel it with the usual precautions. A better way is to take several thousands of the sample, and scorify with a little powdered glass or quartz, repeating, if needful, until the button is of a suitable weight, and then cupel. The bead should be parted, and results must be divided according to the number of thousands used for the assay.

The value of silver lead, in precious metal, is usually reported as so many ounces of gold and so many of silver in the ton, hence an assay ton (Part I), or any quantity commonly used for ore assays, may be taken, being weighed on the pulp scales, scorified

and cupelled, and value calculated as for ore. Or, the fineness in points divided by 10 is the percentage from which ounces or values per ton can be reckoned. One per cent is 291.66+ ounces in a ton; if gold it is worth \$6,029.27.

TO FIND THE VALUE OF A BAR.—The weight of the bar in troy ounces multiplied by the gold fineness (stated in thousandths), gives the weight of pure gold contained, the value of which is computed at \$20.67 per ounce. The weight of the bar, multiplied by the silver fineness (stated in thousandths), gives the weight of pure silver contained.\*

The above values of pure gold and silver, multiplied by the fineness of the bar in each metal (stated in thousandths), gives the value per ounce of such bullion for each metal; and the weight of the bar, multiplied by the respective values per ounce, gives the value of the entire bar for each. It is well to make the calculation in both ways, in order that the one may check the other.

The dealers in assayers' goods distribute, gratuitously, sets of tables showing the value per ounce of bullion of any fineness in gold or silver. The value per ounce being found from these tables, it only remains to multiply the weight of the bar in troy ounces by that.

The value per ounce of bullion of any fineness may be found by means of the "Assay Table for One Assay Ton of Ore" Part I, with more accuracy than by calculation, unless a great number of figures be used.

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\* Silver is reckoned at \$1.2929+ per ounce; its present commercial value is less than half that.

It often happens, in the country, that a bar must be weighed in avoirdupois pounds. One pound avoirdupois is equal to 7,000 grains troy, or 14.583+ ounces; multiplying the pounds and decimal fractions by the latter number converts them into troy ounces. The number of troy ounces corresponding to any number of avoirdupois pounds may be found very simply and accurately from the assay table for 20 grammes of ore, Part I, by proceeding as there directed; or, the value of a bar, of which the weight is given in pounds, may be ascertained by means of the same table without finding the ounces. Again, the value of pounds and ounces avoirdupois may be computed by the aid of the following table, after multiplying the weight of the bar by the fineness, in gold and silver respectively, for the contained weight of the pure metals:—

	GOLD.	SILVER.
1 pound.....	\$301.46.....	\$18.85*
1 ounce.....	18.84.....	1.17
$\frac{1}{4}$ " .....	4.76.....	0.29
$\frac{1}{8}$ " .....	2.38.....	0.14 $\frac{1}{2}$
$\frac{1}{16}$ " .....	1.19.....	0.07 $\frac{1}{4}$

## HUMID ASSAYS OF SILVER BULLION.

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OWING to the uncertainty attending the fire assay of silver bullion, the humid method of Gay Lussac is used in all United States mints and first-class private offices. This method depends on the fact that silver in nitric acid solution is completely precipitated in the form of chloride by a solution of common salt. At the same time the salt is decomposed, so that, if suitable proportions of silver and salt be thus brought together, neither can afterward be found in the liquid.

In the Royal Mint of India, the precipitated silver chloride is dried and weighed, and from its weight that of the pure silver is calculated. In this country and in Europe the quantity of silver in the solution is estimated by the quantity of salt solution of known strength requisite for its precipitation. The salt solution is either weighed or measured; the latter method is the most general, and is called—

**GAY LUSSAC'S METHOD.**—A solution of salt in water is made, of which 100 cubic centimeters will exactly precipitate 1,000 of pure silver, leaving neither salt nor silver in the liquid. This is the *normal solution*. The thousand used in this assay is either 10 grains or 1 gramme, the latter generally. Another solution of salt is made one-tenth as strong as the normal; conse-



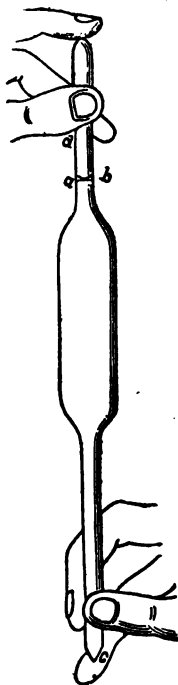
quently 1 cubic centimeter of it precipitates one point of silver. This is the *decime salt solution*. A decime solution of silver is also prepared, of which 1 cubic centimeter contains one point of silver; consequently the two decime solutions are equivalent, volume for volume, each neutralizing the other. The sign for a cubic centimeter is c. c.

c. c.

#### MEASURING THE NORMAL SOLUTION.

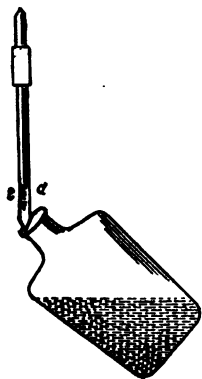
—A glass pipette containing 100 c. c. is used. The accompanying diagram and directions are from Mitchell: "Immerse the jet (c) of the pipette in the solution, apply the mouth to the upper orifice, and draw the liquid into d, above the circular mark, ab. Dexterously apply the forefinger of one of the hands to this orifice, remove the pipette from the liquid, and hold it as represented in the figure. The mark ab is held on a level with the eye and the surface of the solution allowed to descend until it forms a tangent with the plane ab. At this instant the jet c of the pipette is set at liberty by removing the finger against which it had been pressed, and, without otherwise changing the position of the hands, the contents are allowed to run into the bottle appropriated for that purpose [containing the assay],

taking care to remove the pipette as soon as the stream stops."



"If, after having filled the pipette by aspiration, there is any difficulty found in a sufficiently rapid application of the forefinger to the superior orifice to prevent the fall of the liquid below the mark *ab*, the pipette must be removed from the liquid, the orifice being closed by pressing the tongue against it; then apply the middle finger of one of the hands to the lower orifice, remove the tongue, and apply the forefinger of the other hand to the larger orifice, previously wiped dry." The pipette should be rinsed with a little of the solution before being charged, and washed twice with water after use.

MEASURING THE DECIME SOLUTIONS.—The decime solutions are measured in smaller pipettes gauged so that, when filled to the mark *cd*, they allowed 1 c. c. to run freely, the small quantity of the solution remaining in the pipette not forming part of the c. c. The decime salt solution is kept in a bottle closed by a cork which is traversed by the pipette firmly fixed in a hole bored for that purpose. The decime silver solution is kept in a bottle closed by a glass stopper. Quoting Mitchell again:—



"To measure a thousandth ('point') with the pipette, the bottle is held with one hand, and the pipette with the other. The pipette is taken from the solution after its upper orifice has been closed by the forefinger; the lower orifice is then inclined against the edge of the flask to remove the

liquid, which, without this precaution, would remain there; the mark *cd* is then raised to the level of the eye, and, by a suitable pressure of the forefinger on the upper orifice, which may be obtained by giving the pipette a slight alternating circular movement between the fingers, the solution is allowed to run out gradually. The instant the concave surface of the liquid is at the level *cd*, the pipette is firmly closed by pressure of the forefinger on its orifice, which is held above the bottle into which the solution is to be poured, and the forefinger removed so that it can be emptied. It is here necessary to remark that in order to regulate the slow and regular runnings of the liquid from the pipette, by the pressure of the forefinger, the latter ought to be neither too moist nor too dry; if too dry, it will not perfectly close the orifice, even by strong pressure; if too moist, it prevents the entrance of air, and the liquid will not run, or if it does it will be irregularly." (This observation should not be lost sight of in the use of the large pipette.) As it is often convenient to use several c. c. of either decime solution at once, the pipettes are graduated from 1 to 5.

PREPARATION OF THE NORMAL SOLUTION.—One thousand of silver requires  $541.66+$  of salt, hence 1 gramme of silver requires 0.5417 gramme, or ten grains require 5.417 grains. It is more troublesome to prepare pure salt and to measure pure water with perfect accuracy than to make a solution which approaches the required standard, and then rectify it by an addition of salt or water, as indicated by the result of an assay of pure silver, or silver of known fineness, made with it.

The easiest way in which to prepare the approximative solution is to make a *saturated* solution of good table salt, and to dilute a certain quantity of that to the required strength. Take about a pound of salt and dissolve it in about four times its weight of water. Filter the solution, and boil it down to a quarter of the original quantity, or less, causing the deposition of the greater part of the salt. Pour off the liquid, and wash the salt quickly with water. Redissolve the salt in as little water as will suffice, and again evaporate until a little of it is again deposited. Cool the liquid and put it into a bottle. The liquid is a saturated solution containing 26.5 per cent of its weight of salt, as nearly as can be ascertained. One hundred c. c. of this solution, at 60° Fahr., contains enough salt for 49 assays if the thousand be 1 gramme, or for 75.5 assays if the thousand be 10 grains. Therefore, for the gramme system take 1 litre (or 10 charges of the large pipette) of this solution, and add 48 litres of water to make normal solution for 490 assays; or, take 2.08 volumes of the saturated solution, and 97.92 volumes of water. For the grain system take 1 litre of saturated solution to 74.5 litres of water to make normal solution for 755 assays; or take  $1\frac{1}{2}$  volume of saturated solution and 98.66 volumes of water. If more convenient, take for the gramme system 5 parts by any kind of weight, of saturated solution, and 189 parts by weight of water, or 1 to 37.88. For the grain system, 1 part, by any kind of weight, of the saturated solution to 58.89 of water.

If preferred, pure salt may be made by neutralizing

pure hydrochloric acid with pure sodium carbonate or bicarbonate, evaporating to dryness and heating almost to redness; the mass should be powdered and kept in a stoppered bottle. Nearly pure salt may be prepared by dissolving table salt in water, boiling until a large proportion of the salt is deposited, pouring off the liquid, washing the salt twice with a little water, and drying, first between sheets of blotting paper, and then by heating almost to redness. In making normal solution from the dry salt, take for each litre of water 5.417 grammes, or 54.17 grains, according to the weights used for the assays. The most suitable way in which to operate is to place the salt, weighed for the required number of litres of solution, into the litre flask, and add a sufficient quantity of water to dissolve it easily; when the salt is completely dissolved, fill the flask to the mark with water, and empty it into a carboy; again fill the flask, and pour the water into the carboy, as many times as may be necessary to make the desired quantity of solution. Or take 10 pounds avoirdupois weight of water and

for 1 gramme = 1,000 — 24.61 grammes

“ 10 grains = 1,000 — 246.0 grains

of salt; dissolve the salt in a small portion of the water and add the remainder.

The solution, in whatever way prepared, must be thoroughly mixed; this may be done by rolling and shaking the containing vessel, or by stirring with a wand. The plan recommended when dry salt is used, of dissolving it first in a small portion of the water, makes it easier to be sure that it is *all* dissolved than

if it were added to the whole quantity of the water in the larger vessel; the mixing of the solution with the rest of the water is not difficult.

**PREPARATION OF THE DECIME SALT SOLUTION.**—Any volume of the normal, diluted with 9 such volumes of water, makes a decime solution. Measure with the large pipette 100 c. c. of water, which pour into the appropriate bottle; remove 10 c. c. of the water, by means of one of the small pipettes, and replace it by an equal volume of normal solution; mix by shaking; this small quantity is for use in correcting the standard of the normal; after this has been done, a new decime solution must be prepared if the error of the normal was considerable, and a larger quantity will be required. To prepare it measure 1 litre\* of water, or weigh 1,000 grammes at 60° Fahr.; remove 100 c. c. of the water, and replace it by 100 c. c. of the corrected normal solution, by means of the large pipette.

**PREPARATION OF THE DECIME SILVER SOLUTION.**—One thousand of pure silver is dissolved in about 7 c. c. of nitric acid in a litre flask, and the flask is then filled to the mark with distilled water, or at least with water which does not cause the slightest turbidity. If a litre flask is not at hand, 1,000 grammes of water (15,432 grains) may be weighed, a volume of it equal to that of the silver solution removed by means of a pipette, and the remainder added to the silver solution. This quantity will suffice for a very considerable number of assays, and as it will inevitably become stronger

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\*A litre is 1,000 c. c.

by evaporation if long kept, it may be as well to make a smaller quantity; it is only necessary that each c. c. of it shall contain a point of silver and not more.

**STANDARDIZING THE NORMAL SOLUTION.**—A thousand of pure silver is dissolved in about 10 grammes of nitric acid of about 32° Baumé, in a bottle of clear glass which will contain about 200 c. c.; to facilitate the action, the bottle is placed in a vessel containing warm water. When the silver is dissolved, the red fume which fills the bottle is blown out by means of an inserted glass tube so bent as that the workman's face may not be over the bottle while blowing. As soon as the assay has cooled, 100 c. c. of normal solution are added by means of the pipette, and the bottle is put into a japanned tin case, or enveloped in a cloth, to prevent the action of the daylight on the silver chloride, which might slightly vitiate the result,\* and the stopper, previously dipped in pure water, put in. The



bottle, held as shown in the diagram, is briskly shaken for several minutes, which causes the precipitate to curdle, and settle rapidly, leaving the liquid perfectly clear. On placing the bottle on the table, to allow of the settling, a brisk to-and-fro circular movement is given to it, so as to wash down from the sides any adhering chloride. When the liquid is quite clear, the stopper is removed and sus-

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\*This precaution will be unnecessary if the room is illuminated by windows of yellow glass.

pended in a clamp, or bracket, so that the moistened part may touch nothing. A c. c. of decime salt solution is poured into the bottle. If, immediately or after a few seconds, a cloudiness is perceptible in the liquid, it is evident that the normal solution is weak, because 100 c. c. of it have not precipitated 1,000 of silver; the liquid must be again cleared by shaking, and another c. c. of decime salt solution added, and so on until no further cloud is produced. A record of the number of c. c. of decime solution used is kept, but the last one is not counted, because it produced no effect, and the one before the last was probably not all required, so it is counted as a half. Supposing that 7 c. c. caused precipitation, but the 8th did not, only  $6\frac{1}{2}$  must be counted.

It now *appears* as though 1,006 $\frac{1}{2}$  points of silver had been present in the solution, but it is known that there were but 1,000, because that quantity was weighed; the normal solution is therefore  $6\frac{1}{2}$  points weak, and requires a corresponding addition of salt. The quantity of salt required is found by the following—

RULE.—Multiply the weight or measure of salt or saturated solution used in making the entire quantity of normal solution by the number of c. c. of decime salt solution which were *necessary* to complete precipitation, and divide by 1,000. The result is the weight or measure of the required addition.

As a portion of the normal has been withdrawn for the test and for making the decime solution, the quantity thus found may be corrected by the deduction of a corresponding percentage; but it is not worth while, un-



less either many tests have been made, or but a small quantity of normal has been prepared, for even though the addition of salt required may be considerable, it is evident, from the fact that the normal is weak, that the saturated solution used was less strong, or the salt less pure, than was supposed, so that the added quantity will have the same deficiency in proportion. If dry salt was used in making the normal, and consequently will be used also in the correction, the required quantity should be dissolved in some of the original normal poured into a beaker for that purpose, and then mixed with the main stock, the beaker being rinsed with a very little water and that also added. Another test must then be made, and, if necessary, another correction.

If the first addition of 1 c. c. of decime salt solution produces no precipitate, it is known that the normal is either correct or too strong; to ascertain which, add 2 c. c. of decime silver solution; one of these is consumed in neutralizing the c. c. of decime salt previously added, hence only one is counted. Shake down, and add another c. c. of decime silver; if this produces no cloud, the normal is correct within at most a point, and may be accepted, for it is needless to make it perfect, for reasons which will appear hereafter. If a cloud is produced, shake down again, and continue adding decime silver solution and shaking down until the last added c. c. causes no cloudiness; then, as with the salt, reject the last c. c. and half of the preceding one. Supposing that, in all, 5 c. c. have been used, we reject one of the first two added, because it only

counteracted the c. c. of decime salt previously added; we also reject the last, because it gave no precipitate, and the one before the last we count as a half c. c. Thus we have  $2\frac{1}{2}$  c. c. of decime silver solution which were necessary to complete the decomposition of the salt in 100 c. c. of the normal. *Apparently* only  $997\frac{1}{2}$  points of silver were dissolved, but we know that 1,000 were so; consequently the normal is  $2\frac{1}{2}$  points too strong, and water must be added. The quantity of water required is found by the following—

RULE.—Multiply the quantity, by weight or measure, of water used in making the entire quantity of normal solution by the number of c. c. of decime silver solution which were *necessary* to complete the assay, and divide by 1,000. The result is the quantity of water to be added to the normal solution. Mix thoroughly.

A correction for the percentage of the normal which has been used is more necessary in this case; still it is hardly worth the trouble, because, if the error of the normal is large, a second rectification will probably be required, and if it is small, the difference will not be perceived. After mixing, test again; when the normal is sufficiently exact, make a new decime salt solution.

EXAMPLES.—The normal solution was made with 5 lbs. avoirdupois of saturated solution, and a test assay on pure silver required  $8\frac{1}{2}$  c. c. of decime salt solution to finish; then, 5 times  $8\frac{1}{2}$  make 42.5, and this divided by 1,000 gives 0.0425 lbs. of saturated solution to be added to the normal. A pound avoirdupois is equal to 7,000 grains troy, and, multiplying this by 0.0425, we have 297.5 grains.

Again, the normal solution was made with  $189\frac{1}{2}$  lbs. of water, and proves to be too strong, a test assay requiring  $5\frac{1}{2}$  c. c. of decime silver solution to finish; then, 189.5 multiplied by 5.5 gives 1,042.25, which, divided by 1,000, gives 1.04 pounds of water to be added.

If dry salt had been used, or if the saturated solution or the water had been measured by volume, the same kind of calculation would give the required result in terms of the weight or measure used.

When the normal solution is within about a point of being correct, it may be accepted, perfect accuracy being needless on account of the changes of volume produced by alterations of the temperature, for which a correction must be made as hereafter directed, and includes any inaccuracy in the standardizing; for the present, however, the solution will be supposed to be exact.

**THE ASSAY.**—For the sake of convenience, a c. c. of either of the decime solutions will be called a *point of silver*, or a *point of salt*, as the case may be; also 100 c. c. of the normal solution will be called a *thousand of salt*.

One thousand of salt requires 1,000 of silver, and such a weight of the bullion is taken for the assay as contains, as nearly as may be, 1,000 of silver. This being dissolved and 1 000 of salt added, we then proceed, by means of the decime solutions, to ascertain how many points of silver more or less than 1,000 the assay contains. Knowing the weight taken, and having found the weight of the contained silver, it is

easy to find by proportion what weight of silver 1,000 of the bullion contains, which is its fineness.

In order to be able to take the required weight of the sample, we must have an approximate knowledge of its silver fineness; this is obtained by means of a preliminary assay by cupellation, and, if necessary, parting. As we know from experience that there is a certain loss of silver in the cupellation, we add to the result of the preliminary assay such a number of points as the judgment may dictate. From the approximate fineness thus obtained we calculate the weight which must be taken in order that the assay may contain nearly 1,000 of silver; thus, suppose the cupellation (and parting) gave a silver fineness of .864, and we add 3 points for loss, making .867, then, as 867 are to 1,000 so are 1,000 to 1,153.4, and we weigh off 1,154; hence the—

**RULE.**—Divide 1,000,000 by the number of points of approximate fineness; the result is the desired weight in points.

Dissolve that quantity of the sample in about 7 c. c. of nitric acid, in the manner directed for the standardizing of the normal solution; cool, add 1,000 of salt, shake down and add a point of salt. If a cloud is produced, proceed to complete the assay with salt, as in testing the normal solution, keeping a record of the number of points added, finally rejecting the last and half of the preceding one. Suppose 1,154 of the bullion to have been taken, and that 3 points of salt have given a precipitate, the 4th none, we have then found that 1,154 points of the bullion contain  $1,002\frac{1}{2}$

points of silver; to find the fineness, we take the proportion, as 1,154 are to 1,002.5 so are 1,000 to 869.7; as not less than a point is usually stamped on a bar, we may say the bullion is .869 fine.

If the first added point of salt gives no precipitate, add a point of silver to neutralize it, shake down and add another; if this produces no precipitate, the assumed fineness was correct; if otherwise, proceed to finish with silver in the same manner as with salt; then rejecting the first point because it was required to counteract the point of salt, and the last because it gave no result, also half of the preceding one because it was probably only required in part, deduct the remainder of the added points from 1,000, which leaves the number of points in the assay. If, as before, 1,154 of the bullion were taken, and 1 added point of salt gave no precipitate, and 4 successive points of silver gave a precipitate, the 5th none, then it is known that 1,154 of the bullion contain  $998\frac{1}{2}$  of silver, and as 1,154 are to 998.5 so are 1,000 to 865.25, or the bullion is .865 fine. Hence, to find the fineness, the following—

**RULE.**—Multiply the points of silver found by 1,000, and divide by the points of bullion taken.

Greater exactitude can be attained by finishing with half points, but for ordinary purposes it is not necessary. The gold fineness of the bullion is found in the preliminary assay by cupellation and parting.

It is better to finish with salt than with silver, so the weight of bullion taken for the assay should be such as to contain a little more, rather than less than 1,000 of silver; and if it is not so, and is found to contain

less, a number of points of silver may be added at once, so as to make more than 1,000; the assay is then finished with salt. An account must be kept of the silver and salt added, the one + the other —. If more than eight points of salt or silver are required to finish, it is advisable to make another assay on the basis of the result found.

The last trace of precipitate is somewhat difficult to discern; it can be seen best by looking obliquely upward, through the liquid, while holding the bottle toward the light. Another help is to have a black background, for which a black felt hat answers. The writer and some others have found it advantageous to have the assay solution tinged by copper; the white silver chloride shows very plainly in the blue solution. An experienced assayer, on observing that the first added point of salt causes a considerable precipitate immediately, will add several more points before shaking down, being able to judge about how many will be required; he can also tell, almost with certainty, when another point will be without effect.

In case the furnace is not fired, a preliminary assay may be made by means of the blowpipe. It is not necessary that any special weight be taken, therefore select a piece of the bullion which may weigh about 100, more or less; weigh it, cupel it with lead before the blowpipe flame, not allowing the flame to touch the metal, but heating the cupel around it by directing the point first on one side and then on another; weigh the bead, part and weigh the gold if there is much; then as the weight of the silver is to that of the assay,

so is 1,000 to the weight of the bullion to be taken for the volumetric assay, to which a few points may be added for the loss, which is likely to be greater, at least in inexperienced hands than in the muffle. The gold may be calculated for 1,000 on the same principle, but it may be better to dissolve 100 of the bullion in nitric acid, and weigh the gold from that, the only difficulty being that it is likely to be in finer powder than when parted from a bead which has been purified from base metal by cupellation.

An excellent preliminary, and even an accurate assay, may be made by means of a Mohr burette.\* Dissolve 1,000 of the bullion in the usual manner. Charge the burette very accurately with normal solution, and run that into the assay bottle, first in larger, then in smaller quantities, finally drop by drop, shaking down each time, until the last drop or two produces no precipitate. The value of a drop may be ascertained by noting how many drops lower the float 1 c. c. The last addition, which produces no precipitate, is not counted, and the preceding is counted for half its value. The burette should be read after each addition toward the end; it should be of 100 c. c. capacity, graduated to tenths, and furnished with a float. If, by accident, an excess of salt solution is run in, add ten or more points of decime silver solution, making a note of it; and then finish with salt. If preferred, the assay may be finished with decime salt solution from a second burette, or from the small

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\*For a description of this, and its use, see articles on manipulation and on volumetric assays of copper, in Part III, this volume.

pipette, but with care this will not be necessary, as the normal can be measured to less than a point. The number of c. c. necessary for complete precipitation, multiplied by ten, is the fineness, provided no addition of decime silver has been made; otherwise that must be deducted. Special care must be taken that no air remains in the jet of the burette when charged to which end it is best to partly charge it from below by suction, and then fix it in the stand and fill it from above.

**CORRECTING THE ASSAY.**—It has thus far been supposed that the normal solution was accurately standardized, so that 100 c. c. would exactly precipitate 1,000 of silver. In order that this may be the case, it must not only have been made of the correct strength, but it must always be used at the same temperature, which cannot well be. For this reason a *check assay* is made, on pure silver, with each set of bullion assays, a number of which can be conducted at the same time, and are corrected according to the error found, whether that is caused by expansion or contraction of the solutions, owing to a change of the temperature, or from inaccurate standardizing.

The result of an assay of pure silver should be 1,000 fine; if it gives more or less, the assay of each sample of bullion must be corrected in proportion to its fineness. This is arrived at with sufficient exactness by adding to, or subtracting from, the number of points found in the assay, the amount of the error of the check, *before* calculating the fineness of the bullion. Thus: If the result of the check is  $2\frac{1}{2}$  points *above* 1,000 that quantity must be deducted; if below, added.



Suppose the check was  $2\frac{1}{2}$  points high, and 1,154 of bullion gave  $1,005\frac{1}{2}$ ; we deduct  $2\frac{1}{2}$  from that, leaving 1,003, from which we then calculate the fineness, which is 870. If the check had been  $1\frac{1}{2}$  low, we should have added  $1\frac{1}{2}$ , making 1,007 in 1,154, and the fineness would have been .873.

GENERAL REMARKS ON THE HUMID ASSAY.—Silver bullion from retorted amalgam is said to sometimes contain mercury. The presence of mercury in sensible quantity impairs the accuracy of the humid assay as described. To overcome this difficulty, add to the dissolved metal in the bottom about ten times as much of sodium acetate, and then proceed as usual. Mercury may be detected in the assay by the silver chloride remaining white when exposed to the daylight; in the absence of mercury it quickly assumes a purplish hue, finally becoming almost black; if the presence of mercury is detected in an assay, a new assay must be made, adding the sodium acetate before the salt. If the bullion is suspected of containing mercury, the acetate may be used, as it will do no harm in any case, or a little of the bullion may be dissolved in nitric acid, the silver precipitated by salt, and the precipitate exposed to the light; if the precipitate becomes discolored, it contains no mercury.

If a number of assays are to be made, they may all be dissolved at once, and each bottle dosed with 1,000 of salt, shaken down, and then a point of salt poured into each, and so on, saving time.\* When the assays

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\*Sets of bottles, with pointed stoppers, may be bought at the dealers. Also an apparatus mounted on springs for shaking a number at once.

are finished, the bottles should be washed thrice with pure water. Care must be taken, especially in warm weather, not to allow the perspiration to come in contact with the liquids or with the interior of the vessels, because it contains salt. Salt in the water used for making the normal solution is not injurious, but if there is much, it may be allowed for; the decime salt solution should be made with pure water. The nitric acid used must not show the slightest turbidity when pure silver is dissolved in it, nor on the addition of salt, so that the regenerated parting acid, hereafter mentioned, is not suitable for the humid assay; that sold in bottles marked C. P. (chemically pure) requires to be diluted.

In large establishments, the normal solution is kept in an elevated tank, from which a fixed pipette is filled by a tube. The best style of apparatus is that in which the pipette is filled from the lower end, holds exactly 100 c. c., and, when full, is closed at the top by a finger. The beak is then touched by a sponge to remove any adhering drop of solution, the assay bottle is placed beneath it, and, by removing the finger from the upper orifice of the pipette, the solution is allowed to run.

The decime solutions are used by means of hand pipettes, or a Mohr burette. A number of bottles are shaken at once by machinery driven by hand or steam power.

It is not essential that the normal pipette shall contain 100 c. c., it may be much smaller, and the solution proportionately stronger, if the neck is narrowed

in similar ratio so that the measurements may be more exact. The writer has made and used pipettes of which that for the normal solution was formed from the barrel of a small glass syringe, the original nozzle being utilized as the neck, on which the mark was made by filing, and a delivery jet formed on the other end by the aid of the blowpipe; it contained less than 10 c. c., or about 145 grains of water. The auxiliary pipettes were made from glass tubes, with neck and beak like the normal. (The decime solution pipettes are usually made with a beak, but not a neck.) These were of one-tenth the capacity of the normal, instead of  $\frac{1}{10}$ , as is usual, but which would have been inconveniently small in this case, and to compensate this, centime instead of decime solutions were used. The orifices were all very narrow, and the pipettes were charged by sucking, and emptied by blowing. The larger pipette not corresponding to any standard measure, it was filled to the mark with water and emptied into a graduated cylinder as many times as was necessary to make a definite measure, which afforded a basis for making any required quantity of a normal solution of which each pipetteful should contain the proper quantity of salt. To test the accuracy of this apparatus, four separate pipettefuls of water were weighed on a delicate balance; they agreed two and two, the difference between the pairs being  $\frac{1}{10}$  grain, which was closer than was necessary; crucial test assays, the one finished with salt, the other with silver, agreed within a half point. This apparatus is far better to work with by hand than the larger one. It

is necessary to add some water to the assay to furnish the requisite volume of liquid for the settling. A 10 c. c. pipette can be bought for a trifle, and the small ones will be the same as those ordinarily used if centime instead of decime solutions are employed.

The researches of G. J. Mulder have shown that in the humid assay as described, a point may be reached at which *either* salt or silver will give a precipitate, and it has been asserted that in a warm climate this may give rise to an error of several points; there is, however, no danger if matters are so managed as that the check and the bullion assay are both finished in the same manner, preferably with salt, which gives the better clearance of the liquid.

**RECOVERY OF SILVER.**—The assays being finished, the bottles are emptied into an earthen jar, and when a sufficient quantity of silver chloride has accumulated, the liquid is poured off, the chloride is washed, and some pieces of iron or zinc and very dilute sulphuric acid are added. In a few days the silver is reduced to the metallic state in the form of a gray powder which is washed, dried and melted in a black-lead crucible with some borax; as this silver will contain all the gold which was in the assays, it is not suitable for the purpose of inquartation, nor for humid assay checks.

**PREPARATION OF PURE SILVER.**—Dissolve the thoroughly-washed chloride from the humid assays, or from the parting acid treated with salt or hydrochloric acid, in warm, strong brine of common salt. Filter the solution into a porcelain or glass dish (a wash basin will serve). In the middle of the dish place a

smaller porous vessel containing salt brine, with a very little nitric acid. (For a porous vessel a Hessian crucible, which has stood full of water until soaked through, will answer.) Cut a piece of sheet copper of such a size as to form a cylinder which can be placed in the porous vessel, not touching the side. From one edge of the copper cut a narrow strip, but not entirely off, leaving it connected to the sheet. Take a strip of test silver long enough to form a ring around, but not touching the porous vessel, and from the edge cut a strip partly off, as with the copper. Clean the copper by immersion for a few minutes in dilute nitric acid and then in water. Connect the copper and the silver by soldering the ends of the strips, or by folding them together. Bend the plates suitably and place the copper in the porous vessel, the silver in the basin containing the solution of silver chloride, the connecting strips forming an arch above the liquids. Keep the whole warm, and, if necessary, renew the solution in the porous vessel, or add a little acid from time to time.

Pure silver in the form of small white crystals will be deposited on the silver plate. Iron or zinc in the porous cell would work faster than copper, but would not give pure silver, as they would cause the precipitation of any trace of lead, copper, etc., which might be present, as well as of the silver. If pure silver for a plate cannot be had, a piece of coke may be used instead, being connected with the copper by means of a copper wire soldered to that and connected with the coke by being inserted in a hole which is then filled with melted lead. This connection must be kept out of the liquid.

The precipitated silver should be washed, first with hot water containing some salt and a *little* hydrochloric acid, and then with pure hot water until that gives no precipitate on addition of solution of silver nitrate. If destined to be melted, the silver may be dried by evaporation after draining; otherwise it should be first pressed between sheets of filter paper, and then dried by heat, and kept in a stoppered bottle.

The dried silver may be melted in a clean black-lead pot, or in a clay crucible lined with charcoal, a little borax being added, and cast into narrow bars like those of solder. The bars may be cut into chips by the chisel, or laminated by the rolling mill, after being reduced to a thickness of about  $\frac{1}{4}$  inch by hammer and anvil, being cut into suitable lengths, and annealed whenever necessary for the rolling. The filter, through which the solution of silver chloride in brine was passed, may be dried, dressed with litharge and borax, smelted in a crucible, and the resulting lead button cupelled for recovery of gold.

To line a crucible beat in, little by little, finely-ground and slightly-moistened charcoal until the pot is filled, then scoop out the central portion, leaving a lining about half an inch thick, more or less; smooth the interior by means of a glass rod or a test tube.

RECOVERY OF ACID.—The nitric acid used in the humid assays is so far saturated with silver that its recovery would scarcely be profitable, especially as it is contaminated by sodium nitrate, resulting from the salt used in the assay. That which has been used for parting, however, contains but little silver in propor-

tion, yet it is usually wasted, because salt is used to precipitate the silver and the acid is rendered unfit for further use in assaying. The acid, as well as the silver, may be recovered in a suitable condition for reuse, the one for parting, the other for inquartation, by proceeding as follows:—

After standing undisturbed for several days, to allow any suspended gold to settle, the acid is carefully poured or syphoned into a suitable vessel of glass or porcelain, all sediment being left behind, and to it hydrochloric acid is added, little by little, to precipitate the silver as chloride, with great care not to use an excess. The liquid is briskly stirred by means of a glass rod, and then allowed to remain undisturbed until the silver chloride has settled; or, a little of it may be taken in a bottle, cleared by shaking as in the humid assay, and tested with a drop of dilute hydrochloric acid. It is not best to attempt the *complete* precipitation of the silver, as a small quantity remaining in solution will not be injurious, while an excess of hydrochloric acid will. Should the latter be accidentally added, the error can be rectified by means of some silver solution, for which purpose a portion of the original should have been set aside. When a drop of dilute hydrochloric acid produces only a very slight cloud, the liquid is allowed to stand undisturbed for 24 hours, covered to exclude light and dust; it is then filtered through fibrous asbestos, ground bottle glass, or quartz, until it is perfectly bright; it should now give a slight, yet distinct, precipitate on addition of dilute hydrochloric acid to a little in a test-tube, but none with solution of silver nitrate.

The hydrochloric acid produces silver chloride, liberating nitric acid, and the recovered acid is as good as any for the purpose of parting, but should not be used for the humid assay, because a trace of silver is left in it as a safeguard against the presence of chlorine. The presence of a very little copper resulting from previous partings and not removed by this treatment does no harm.

This method was introduced by the writer in an office where a large business was done in gold bullion, and was used for years with perfect success and considerable economy. Ordinary commercial nitric acid, costing about one-tenth as much as the c. p., but often containing a little chlorine, may be rectified by the addition of a slight excess of silver nitrate, and used with perfect safety for parting.

The silver chloride precipitated from the parting acid is treated as described under *Recovery of Silver*, and is again used for inquartation of gold assays; it must contain no gold, and will contain none if the preceding directions are followed.

**VOLHARD'S METHOD.**—The silver is precipitated from the nitric solution by a solution of sulphocyanate of potassium, or of ammonium, generally the latter; it must be pure, or at least must contain no chlorine, and the solution must be made with distilled water.

One part of the silver requires for its complete precipitation, 0.704 parts of ammonium sulphocyanate, but, as this salt is usually a little moist and attracts moisture from the air during the weighing a slightly



larger proportion is taken for the preparation of the solution. In practise it is found that eight grammes, dissolved in one litre of water, yield a solution of which about 100 c. c. will precipitate 1 gramme of silver. If grain weights are used, 80 grains of the salt to a litre of water give about 100 c. c. to 10 grains of silver. As the precipitate is voluminous, it is usual to operate on half the above quantities of metal.

For the purpose of description it is assumed that the gramme system of weights is employed; those who prefer the grain system can use about one-third less of acid, etc., while the figures for the weight of metal will be the same, with the decimal point moved one place toward the right hand.

The solution is standardized, or titrated, as follows: Dissolve 0.5 gramme=500 milligrammes, of test silver in 10 c. c. of nitric acid of about 32° Baumé in a flask of 400 c. c. capacity. Boil gently until fumes cease. Add 200 c. c. of cold distilled water, and, when the assay is quite cold, add also 5 c. c. of a cold saturated solution of iron alum. If the color of the iron salt becomes apparent, add a little more nitric acid which has been boiled immediately before.

Now run in the precipitating solution from a charged Mohr burette, which is graduated to one-tenth, or, at least, to one-fifth c. c., and furnished with a float. At first only a white p. p. ensues; later, each drop produces a red cloud, which disappears on shaking. When nearly all of the silver is down, the p. p. collects in flakes, and the liquid becomes clearer as the end approaches.

When all of the silver is precipitated, a further addition of the precipitant produces a brown color, which does not disappear on shaking. This denotes that the assay is finished, and the number of c. c. of the precipitant consumed, minus half of the last addition, constitutes the standard, or titre, of the solution for the taken quantity of silver, and should be very nearly 50 c. c.; otherwise, the solution may be strengthened or weakened to the desired approximation, but, as explained in the description of the salt solution for the Gay Lussac method, perfect accuracy is needless. The standardizing should be repeated from time to time, also whenever an assay must be made at a temperature which differs much from that at which the last determination was made.

The solution may be run in freely until the red color begins to disappear but slowly on shaking, then only small quantities must be added at once, and finally a few drops until a permanent color ensues. On reading the burette, the half of the last added quantity must be subtracted, because the permanent color proves that there is a slight excess of the precipitant. In order to do this, the operator must ascertain how many drops the burette delivers to the c. c., which will usually be from 20 to 30, depending on the fineness of the jet. If 20 drops equal 1 c. c., 2 drops are one-tenth c. c. A burette which is graduated to fifths can be read to one-tenth with sufficient accuracy, and if, for example, the last addition was 4 drops, the last indicated tenth must not be counted.

If preferred, for greater accuracy, the use of the nor-

nal solution may be stopped near the end of the operation, when the float is exactly on one of the graduations, and the assay finished with a decime solution from another burette, or by means of a one-tenth c. c. pipette, as in the Gay Lussac assay. A decime solution is made by mixing one measure of the normal solution with nine measures of distilled water.

To assay a sample of bullion, take 500 milligrammes of it, proceed exactly as directed above, and divide the result by the titre of the solution. The quotient is the fineness of the bullion.

EXAMPLES.—The titre of the solution is 50.7 c. c., and 500 milligrammes of the bullion require the same quantity to precipitate. In this case it is clear that the bullion is pure silver; this will not often occur. But suppose that 500 milligrammes of the bullion require 48.3 c. c. of solution: then,

$$50.7 \text{ ) } 48.30 \text{ } \underline{.952}$$

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the bullion is .952 fine, and if we wish to be more exact we may carry the division further and stamp .952½. The method of calculating is the same if the titre of the solution is exactly 50 c. c. or below that.

When the bullion contains as much as 70 per cent of copper (or less in unpractised hands), the brown or red tint indicating the ending, is masked by the blue color of the assay. In such a case it is usual to take only 250 milligrammes of the bullion, and an equal weight of pure silver, dissolve, etc., as usual, calculate the result, and deduct .500 for the added silver; the remainder, doubled, is the fineness of the bullion.

EXAMPLE.—The titre of the solution is 49.8 c. c., and 250 milligrammes of the bullion, with 250 milligrammes of silver added, required 29.6 c. c. to precipitate; then,

$$49.8 \overline{) 29.6} \text{ } \begin{array}{l} \text{ } \\ \text{ } \\ \text{ } \end{array} \begin{array}{l} .59437 \\ * * * \end{array}$$

We carry the division far, in this case, because of the final multiplication.

	.59437
Deducting for added silver .....	500
we have.....	<u>09437</u>
and multiplying by.....	<u>2</u>
we obtain the fineness.....	18874

which would be reported as  $.188\frac{1}{2}$  or  $.188\frac{3}{4}$ , according to circumstances.

In making this assay, the following precautions are important:—

1. All nitrous acid must be expelled from the assay, by boiling, before attempting the precipitation, and any nitric acid which may be added afterward must be previously purified by boiling.

2. The assay must not be exposed to the direct rays of the sun.

3. The assay must be completely cooled before the precipitation is begun.

4. The sulphocyanate solution must not be exposed to light more than is necessary, nor to the direct rays of the sun at any time.

Bullion which contains mercury cannot be assayed by this method. (The manner of detecting mercury

in the bullion is given in the description of the Gay Lussac method.)

Palladium gives a false result if present, and cobalt interferes by the color which it imparts to the assay; but neither of these metals occurs in ordinary bullion.

The object of adding silver to an assay which contains but little is to insure the production of a sufficient quantity of p. p. to curdle well, and so clear the liquid. The added silver must be accurately weighed, as any error in the weight would give a double error in the result where only 250 milligrammes of the bullion are used. For my part I think I should prefer the Gay Lussac assay for bullion containing much copper, or cupellation may be resorted to, and then the Volhard method affords a convenient means of making a rough and rapid preliminary as a guide to the quantity of lead required for clearance, and for regulating the composition of the check. But Volhard's method is very convenient and accurate for high-grade bars; it has the advantages of requiring no preliminary, and but little shaking.

As to the calculation, it is probably preferable to take such a weight of the bullion as would correspond to the titre of the solution, and which would be uniform for any number of assays, so long as the standard of the solution should remain the same. Such a weight is found readily by proportion. Suppose the standard of the sulphocyanate solution is 49.8 c. c. to 500 milligrammes of silver, then,

$$49.8:560::50:502$$

that is, 502 milligrammes saturate 50 c. c. of that solution.

Now, if we look upon 502 milligrammes as constituting, for this case, 1,000 "points" of silver, and 50 c. c. of that solution as 1,000 points of sulphocyanate, it is clear that the number of c. c. used in an assay of 502 milligrammes of bullion need only be multiplied by 20 to equal the points of silver in the assay; and the points become thousandths by prefixing a dot.

EXAMPLE.—The titre being as above=49.8 c. c. to 500 milligrammes of silver, or 50 c. c. to 502 milligrammes. Suppose we have three bars to assay: We weigh 502 milligrammes from each sample; and,

No. 1 requires of solution 39.4 c. c.=788 points=.788 fine

" 2 " " " 47.3 " =946 " =.946 "

" 3 " " " 24.7 " =494 " =.494 "

In short, we have only to double the c. c. to obtain the figures which, with a dot prefixed, denote the fineness.

And then, suppose we have a bar which contains a great deal of copper, we take  $502 \div 2 = 251$  milligrammes, add 251 milligrammes of silver, and consume of solution

31.9 c. c.=638 points

deduct for added silver..500 "

leaving, in assay.....138 " to 251 mgrs. of bullion

multiplying by..... 2

276 " =.276 fine.

In adjusting the float to zero, and before reading the burette, time must be allowed for the liquid to drain down the side, which will raise the float a little. The plan of finishing with a decinormal solution may

please those who do not like the finishing by drops, but I doubt if it assures greater accuracy. The accuracy of the determination is limited by that of the measurement of the normal solution by means of the burette, and it is useless to follow that by more delicate means. Owing to the width of the burette, the measurement cannot be as close as by the large pipette in the Gay Lussac method, which has a very narrow neck; however, it is near enough for practical purposes generally, and a closer approximation may be got by doubling the quantity of metal and working to 100 c. c. instead of 50; or, the Gay Lussac apparatus and system can be used with this solution, a preliminary assay being made with the burette.

## CONCLUSION OF PART SECOND.

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ASSAYERS of bullion who work for custom must remember that they are responsible for the value of a bar as stamped by them. If any U. S. mint, or any private office in good standing, finds the value to be less, a *reclamation* is made by, or on behalf of, the purchaser of the bar, which the assayer on whose stamp it was bought must pay if he wishes his stamp to pass in the market; it matters not that his assay may have been correct, the purchaser knows nothing of that. If the chips have been preserved, as they should be, it may, in some cases, be possible to bring the fault home to the office in which it was committed, and the first assayer, if right, may recover his money, but this will hardly avail with the mint, perhaps because "the king can do no wrong." The system pursued in the mints is such as to render mistakes almost, though not quite, impossible. An instance is within the knowledge of the writer in which a reclamation was made on a mint certificate. The error was proved to have been made in the mint, beyond reasonable doubt, but the private assayer had to suffer the loss. It is needless to say that if a bar is stamped too low no complaint is usually made by the purchaser, though the fact, if known to the seller, might lead to a reclamation on his part, and would certainly injure the reputation of the assayer. The inference to be drawn from these remarks is obvious.





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## PART THREE

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## PART III.

### INTRODUCTION.

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THE directions given in Part I for the sampling of ores apply equally to Part III.

In a book of this size it is not possible to give all the methods known for the assay of each metal, nor even all of the modifications by which time and labor may be saved in particular cases.

The aim has been to give one or two processes for each of the substances treated of, which would cover any probable case, and yield results nearly enough exact for general practical purposes in these Western countries, not seeking the highest attainable approximation to accuracy by costly and elaborate methods and apparatus.

Only in a few instances are given special directions for the assay of alloys. In general, the methods given for ores will answer for alloys also, though simpler processes might be used in many cases; to describe these would carry me too far for my design. Those who have much to do with alloys, or who have constant work in special lines, should consult other books.

This book is not designed for the use of college students, scientific men, nor specialists, but as a handbook for ordinary workers.

## MANIPULATION, ETC.

A FILTER must be circular in order that, when placed in a funnel, the edge may be even; and of such size that the edge will be a little below that of the funnel.

To cut a filter to fit a funnel, fold the paper twice in diametrically opposite directions; place it on a table; put the point of a pair of pencil compasses close to, not on, the corner; with a little less than the diameter of the funnel between the points of the compasses, describe an arc of a circle on the folded paper; cut with scissors to the mark. Or, invert the funnel upon the folded paper, the rim at the corner; look down through the stem and see the edge of the paper passing through the center of the funnel; make a mark on the paper where the rim of the funnel intersects the edge. Do likewise on the other edge and again in the middle and at as many points as may be chosen, marking the paper at a distance from the corner equal to the funnel's diameter; cut a little within the marks. Or, fold the paper three or four times; cut straight across at a distance from the corner equal to the diameter of the required filter, then trim to an arc of a circle. Open the twice-folded paper so that it shall form a cone having one thickness on one side and three on the other.

Place the cone in a funnel; hold it with the thumb inside and the fingers outside of the funnel, and wet the paper by a jet from the wash bottle. The filter should fit closely to the funnel all over except at the point, but especially at the rim, which should be air-tight. Press the folded edges with the rounded end of a glass rod, a test-tube, or the thumb nail; also press out all air bubbles from between the funnel and paper.

The size of a filter should be adapted to the bulk of the precipitate when that is wanted, but when a large volume of liquid is to be filtered simply to clear it, a large filter is the more suitable. Filters already cut to a circle and of various sizes and qualities can be bought. For solubility assays of roasted ore, and wet copper assays, gray paper answers and works fast; for lead, zinc, and sulphur assays, a finer quality is more suitable.

In order that a filter may fit properly, the outline of the funnel, except the neck, viewed sideways, must form a triangle with three equal straight sides;\* the worst funnel for this purpose is one that is vase shaped; the side and the neck should form an angle, not a curve.

When a substance is to be weighed on the filter, the filter must be counterpoised by another, or its weight must be known, in order that the net weight of the substance may be obtained. The filter should be dried prior to use, at that temperature at which the substance is afterward to be dried on it, generally the

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\*It is possible, by suitably folding, to adapt a filter to a funnel of which the angle is more or less than 60 degrees.

heat of the water bath, and, after cooling, it must be weighed and its weight noted, or it may be adjusted by clipping to equality of weight with another of the same kind similarly dried.

Where much of this kind of work is to be done, a number of filters may be dried at once and kept under a bell-glass, or in any closed receptacle, in company with an open dish or beaker containing strong sulphuric acid.

The drying of the filters before equalizing the weight, when one is to be used as a counterpoise to the other, is only necessary when very fine work is to be done; in many cases it suffices to take two filters from the same package, or to cut them from the same sheet, and equalize their weight by the aid of the scissors and balance. The counterpoise filter must, in all cases, be exposed to the same heat as the one containing the substance during the drying of that, and the two must be allowed to cool together before the weighing; in a particular case they should be cooled under a bell-glass in presence of sulphuric acid, which will prevent the accession of atmospheric moisture. The same may be said of substances which are to be weighed otherwise than on a filter. In strict analyses the substances are inclosed between two watch-glasses for the cooling in the dessicator, then weighed, glasses and all; but this is scarcely necessary in merely commercial assaying, except with substances which absorb moisture rapidly from the air.

In filtering, fill the filter often, not waiting for it to drain. In washing the substance on the filter, and the

filter itself, let it drain before each fresh addition of water.\* If the quantity of the washings is immaterial, it is best to quite fill the filter; otherwise direct the stream from the wash bottle upon all parts in succession, especially around the rim, which should never be found stained with the filtered liquid when dried. The matter on the filter may generally be stirred up by the stream with advantage to the washing; a bulky, gelatinous precipitate may be carefully stirred with the rounded end of a glass rod; at last, if the precipitate is to be used, wash it down into the point of the filter. Hereafter, to save time and labor, a precipitate will often be designated by the sign p. p.

It is generally best to let a p. p. settle before filtering; then filter the liquid first, as far as practicable. In some cases water is added to the p. p.; stirred, and sometimes boiled and again settled and filtered off, even several times. Finally, allow the p. p. to pass to the filter, washing it cleanly from the vessel by a jet of water, aided if necessary by a brush formed of a clipped feather. As a general rule, in pouring from a vessel, hold a glass rod to the lip, and let the fluid run down the rod and against the side of the filter or other receiver. The lip of the vessel may be greased on the outside with tallow, to prevent loss of the contents by dribbling while pouring out.

The liquid from the filter (*filtrate*) must not be allowed to fall splashing into the receiving vessel, but should trickle down its side; even if the filtrate is not

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\* There are some exceptions to this, as in filtering and washing some sulphides, which must be kept covered to prevent access of air.



required, a neat workman will not have it splashed about the table.

There are several ways of ascertaining when the washing of a p. p. is completed; if the filtrate was acid, the washings must cease to redden blued litmus paper; \* if alkaline, they must not discharge the color of reddened litmus paper. If the water used be pure, a drop from the filter, evaporated on a piece of glass or platinum foil, should leave no film, or only a slight speck.

To dry a p. p. first let it be well drained, which may be aided, in some cases, by sucking the stem of the funnel, but not too forcibly lest the paper be ruptured, or by tapping the funnel. The filter with the p. p. may be carefully removed from the funnel, unfolded, and laid on a warm surface, better with several thicknesses of paper under it; special absorbent tiles are also used; the drying is then completed in the air or water-bath, etc. Or, the filter with its contents may be dried in the funnel, that being placed in the upper end of a conical tube standing on a sand-bath. The drying should be continued until two weighings after thorough cooling have given the same result, that is, if the substance is to be weighed without further treatment.†

To remove a dried p. p. from a filter, invert that on a sheet of glazed paper, or over a porcelain dish, etc.,

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\* Except when directions are given to wash with acidulated water, etc.

† If a p. p., supposed to be dry, is placed on the balance and counterpoised while still warm, and is then found to become lighter, it is certainly not dry. Time may be saved by acting on this hint.

and manipulate it with the fingers to loosen the p. p. Scrape it with a wooden or horn spatula; double it and rub the surfaces together; lay it flat and brush it with a rather hard brush, etc.; according to circumstances.

To remove a wet p. p. from a filter, the way I prefer, in general, is this: Drain, remove from funnel, unfold, place against inside of large beaker, to which, being wet, the filter will cling, while it may be prevented from slipping down by a finger, for which reason the clean part should be on the rim of the beaker; wash by the wash bottle, aided if necessary by a soft clipped feather, using as little water as possible, if that is important; the water may be evaporated off if necessary for further treatment of the p. p. A common method is to pierce the filter by a pointed glass rod, and wash the p. p. through the hole, but after all it is generally necessary to remove and unfold the filter in order to complete the transfer.

When a p. p. is to be dried and weighed on the filter, I sometimes proceed thus: Cut from a sheet two filters of equal size, and adjust them to equality of weight by means of the balance and scissors; place the one filter accurately within the other, and *both* in the funnel; when the filtration and washing are completed, the filters and p. p. are dried together, and in the weighing the empty filter is put on the weight pan; in this way any change of weight which the paper may sustain from the action of the filtrate will affect both filters alike. However, this must not be done when the p. p. is of such a nature as to be liable

to passing through a filter, because if a little should pass through the first and be retained by the second, a doubled error would result.

Another device is as follows: Instead of cutting two filters of equal weight, let the one be heavier, and let it counterpoise the other plus a small piece of filter paper; collect the p. p. on the smaller filter, and, having removed it from the beaker, or other vessel, as cleanly as possible by means of the wash bottle and a clipped feather or rubber-tipped rod, wipe the beaker, and the stirring rod if necessary, with the extra piece of paper, held in a pair of forceps, and put the paper into the filter with the p. p., then wash, etc. This is convenient for a p. p. such as lead chromate, which clings to the vessel, and which cannot be conveniently collected by re-solution and precipitation of the clinging portion.

When a filter is to be burned, the best way, in general, is as follows: Dry; remove the p. p. or residue; fold the filter in the form of a roll or prism, the clean part enveloping the part on which the p. p., etc., rested; wind spirally around it a piece of fine iron, or, better, platinum wire, leaving an end of wire projecting to serve as a handle, touch the roll to a flame, and hold it over a dish until it is burned to ashes; it may be touched several times to the flame if necessary. The wire will retain the ashes until combustion is completed, then a slight tap will cause them to fall into the dish. The wire may be brushed to remove any adhering particles of ash.

A filter may also be burned in a porcelain dish, in a

muffle or over a flame, but the preceding method is better generally.

In some cases a p. p. may be collected without a filter, *e. g.*, lead sulphate, which, if heated, settles well; the clear liquid is drawn off by sucking it into a pipette; then pure or acidulated water, etc., as the case may be, is added, the p. p. is stirred, again settled, and the liquid removed as far as possible, and so several times. Finally the p. p. is dried by evaporation in a tared or counterpoised vessel.

A method which is not so often used as it might be in cases to which it is applicable, is to dissolve the washed p. p., whether on a filter or not, receive the solution in a suitable tared vessel, dry by evaporation, ignite if requisite or admissible, and weigh in the dish. The substance must be of such a nature as to admit of its recovery from the solution, by evaporation, in a pure state, and in the original or in some other suitable form. Some substances can be dissolved in boiling water, others in ammonia or a volatile acid, etc.

To use a Mohr burette (see volumetric copper assays), fix it in a vertical position; in reading it, if there is no float, the middle of the concave surface of the liquid is the indicator; the eye must be level with that. With a float, the mark around that is the indicator; and the eye must be level with it, so that it appears as a line, not an oval. In charging the burette, first fill it, and see that no air bubbles remain, open the pinch-cock, and allow the liquid to escape into a vessel beneath until all air is expelled from the neck, then refill and again let the liquid flow until the indicator is exactly

coincident with the upper mark; the burette is then ready for use.

A draught place should be provided in which acids may be boiled and hydrogen sulphide used while the fumes are conducted away by a flue. Such a closet is conveniently made by fitting a box on the outer side of the lower half of a window, so that the lower sash forms a front which can be opened or closed, and through which the progress of the work may be observed; it is still better if the back be glazed also; in the top or roof is a flue which, if convenient, may be connected with a chimney. Within the closet the coal-oil stove, lamps, etc., are placed.

When a beaker is used for boiling, it should be placed on a sand-bath, or a piece of wire gauze should be interposed between it and the lamp flame. For a cover nothing is so suitable as an inverted funnel, of such a size as to fit within the rim of the beaker or dish; it allows fumes to escape freely, prevents loss by effervescence, etc., and the liquid which forms on its side by condensation runs down the side of the vessel and prevents incrustations forming there.

When a dish is used on a coal-oil stove, place under the dish a piece of tin plate having a hole in the center suited to the size of the dish, so that the principal heat may reach the bottom, while the sides are protected; this prevents some risk of cracking the funnel used as a cover, although that has never happened in my practise; it also tends to prevent any change in the constitution of a salt which may form an incrustation on the side of the dish. In some cases, however, when the object is to evaporate rapidly without boiling, this

precaution, as well as that of a cover, may be dispensed with, though it is then often better to use the water-bath. A dish is often covered during an evaporation by a sheet of filter paper sustained by a glass rod.

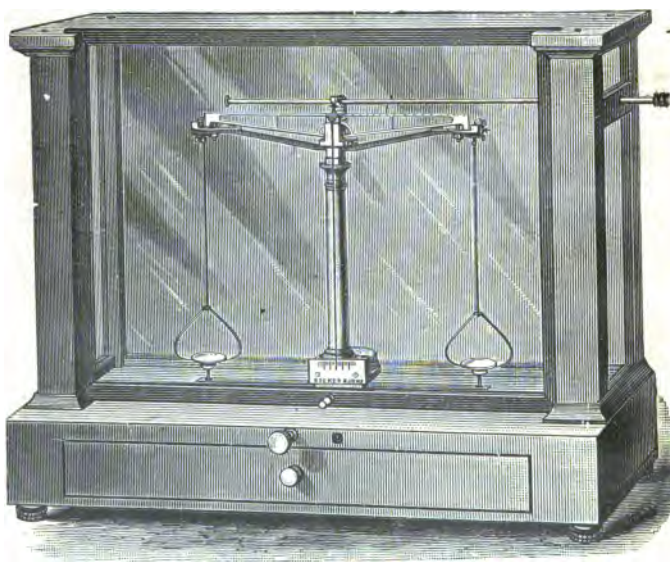
Hydrogen sulphide is a gas which has the odor of rotten eggs; it is produced by the action of dilute sulphuric acid on ferrous sulphide, which may be made by melting iron filings or borings with sulphur, or may be bought. The gas is conducted, by rubber and glass tubing, into water to wash it, and then into the liquid treated; it is used in certain wet assays to precipitate certain metals from solution; various kinds of apparatus for its production can be bought. For all the purposes of this book an eight-ounce generator will suffice.

Beakers may be had with the edge ground, also covering plates of ground glass which form an air-tight joint if the edge of the beaker is greased with tallow. A beaker may be covered by a piece of moist filter paper, which is pressed around its edge and which adheres when dry. Such tight covers are needed when hydrogen sulphide has been applied and the p. p. requires much time for settling.

Wash all vessels after use. Balls of wet paper are useful for cleaning the interior of matrasses, etc. Test-tubes sometimes require a swab on a slip of wood, or a test-tube brush. To rinse a matrass, flask, bottle or tube, put some water in it, stop the mouth with the thumb, invert the vessel, shake it so as to wash all matter down to the mouth, then remove the thumb. Warm water, with a little hydrochloric acid, will remove iron oxide and many other residues. Vessels

which are to be used for parting, for the humid assay of silver, or for any other purpose in which a trace of a chloride or chlorine would be objectionable, must be rinsed three times with pure water. Vessels which have been used in the copper assay, etc., with potassium xanthate, are best cleaned by a solution of potassium cyanide followed by water. The clippings of filter paper are useful for drying glassware.

The motto of a workshop should be, "*A job is not finished until everything is put in order.*"



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NOTE.—The assay balance shown by accompanying cut is strong enough for the weighing of most of the wet assays, while also delicate enough for that of beads from gold and silver ores. Lead and copper, etc., buttons may be weighed on the pulp scales.

## LEAD ORES.

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**FIRE ASSAY.**—The fire assay of lead is not accurate; the result is always too low unless the button is contaminated by other metals; yet it is the method which smelters accept as a basis for the purchase of ores. The assay is like that of gold and silver ores by the second crucible system (see Part I), except that litharge is not used.

A half A. T. of ore is a convenient quantity, but, if fractional A. T. weights are not at hand for the weighing of the button, 100 or 200 grains, or 5 to 10 grammes, will be found suitable; the result is always reported in percentage. To ores which contain copper oxide or carbonate, and not enough of sulphur to combine with the copper so as to keep it in the slag, some sulphur may be added for that purpose; or, the weighed assay may be boiled in nitric and sulphuric acid, which will dissolve the copper, leaving the lead as insoluble sulphate; the liquid must be diluted with water and filtered off, the residue dried, dressed, and smelted for the lead.

A general formula for the dressing of lead ores is,

Ore.....	1 part
Soda.....	2 to 3 parts
Flour, about.....	$\frac{1}{10}$ part
Borax, about.....	$\frac{1}{2}$ “



mixed in a crucible. Add several 12d nails and a liberal cover of salt. French pots are the best to use unless the pot is to be broken for extraction of the button, which is only necessary with very poor ores; in other cases the assay may be poured into the mould.

Melt the assay, uncovered and with care, until frothing ceases, then cover, but not quite closely, and increase the heat to very bright redness. With sulphuretted ores, and especially with complex ores, a high heat must be kept up for 20 minutes after frothing has ceased under a moderate heat. With a clean ore consisting of lead carbonate with probably some sulphate, less time is requisite, but, in any case, the assay is not finished until the nails can be removed free from adhering lead after washing them in the slag and tapping them against the side of the pot; the slag must be quite fluid. Arsenic, if present, will form a separate button of iron arsenide, which should be hard and brittle. If there is much antimony in the ore, it, or a part of it, will go into the lead, giving a hard or brittle button and a false result which must then be corrected by the wet way. Clean lead may be got from antimonial galena, by melting with a plenty of soda, omitting iron and flour.

Pure carbonate of lead does not require the nails if enough of flour be used, yet I prefer to employ nails with all ores lest there may be some sulphate, etc., present.

The lead button may be cupelled for precious metals after the weighing, and the weight of those found deducted if worth while. Rich carbonate ores will yield

all of their gold and silver; in other cases an approximate assay for the three metals may be made by adding to the dressing such a quantity of granulated lead or litharge as may be required to produce a suitable button with the lead from the ore; the lead or litharge added must be weighed, and a corresponding quantity deducted from the button weight, in order to determine the percentage got from the ore. In case litharge is used, it must be assayed for lead, because commercial litharge is not pure; it sometimes contains only 85 per cent of its weight of lead, although pure lead oxide contains very close to 93 per cent. Rich, clean ores, without antimony, may be assayed by fusion with 3 or 4 parts of potassium cyanide under a salt cover; this may be done in a scorifier in the muffle, as a high heat is not needed and should be avoided.

The lead buttons are cleaned by hammering and brushing; they must be malleable and must not show red spots or streaks of copper; otherwise, either the entire button or a weighed portion of it must be digested in hot dilute nitric acid, which will dissolve lead and copper, leaving antimony (and tin) insoluble. The solution must be filtered, and the lead precipitated by sulphuric acid and determined as directed in the wet assay; then, as the weight taken is to the weight of pure lead found, so is the weight of the entire button to that of the lead therein, from which the percentage is to be reckoned for the weight of ore assayed.

WET ASSAY.—Attack a weighed quantity of the powdered and dried ore with a mixture of nitric and sulphuric acids (a little hydrochloric acid may be added

if necessary), in a covered casserole or porcelain dish. Boil until the ore is decomposed and until white dense fumes of sulphuric acid are evolved while still some liquid remains (otherwise add some sulphuric acid). Cool; add water cautiously; digest warm for a short time; filter, and wash with water to which a few drops of sulphuric acid have been added.\* After draining, remove the filter, with its contents, from the funnel to a porcelain dish or a beaker; unfold and lay it flat on the bottom; cover it with ammonia and add acetic acid until the mixture distinctly reddens blue litmus paper. Keep warm for half an hour, stirring occasionally. Decant the liquid upon a filter; wash the residue in the dish several times by decantation, adding to the water at first a little ammonium acetate made by mixing ammonia and acetic acid, with care that the acid predominates; finally transfer all, including the original filter, to the filter and complete the washing; the filtrate contains the lead in solution and may also contain lime, not baryta nor strontia. To the filtrate add dilute sulphuric acid as long as it produces a p. p. Heat; settle; filter; † wash with sulphuric acidulated water, finally, once or twice, with pure water, or, better, with alcohol.

The lead is now on the filter in the form of sulphate, as it was after the first filtration, but freed from impurities. Dry it on the filter; transfer it to a small por-

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\* If copper is present, it may be determined in the filtrate. See Assaying Copper Ores, Part I.

† Or wash in the weighing vessel as directed in the article on manipulation.

celain crucible of known weight (or counterpoised); burn the filter; add the ashes to the p. p. and moisten them with a drop of sulphuric acid; heat nearly to redness; cool and weigh in the crucible. If very particular, burn another filter of equal weight with the first, moisten the ashes with sulphuric acid, dry, and put them on the weight pan when weighing; \* a convenient way in which to manage this is to let the counterpoise, or part of it, be another crucible in which the second filter may be burned and the ashes treated as described.

Unless for very particular work, the lead sulphate may be weighed on the (tared, etc.) filter, after drying at 248° Fahr. (in an air-bath). The lead sulphate contains 68.32 per cent of lead.

A modification of the process is as follows: Treat the ore with acids as above; wash thoroughly with sulphuric acidulated water, so as to remove all lime; dry the insoluble matter, consisting of lead sulphate, silica, barium sulphate, etc., and weigh it; extract the lead sulphate by means of ammonium acetate as above; wash, dry and weigh the residue. The loss represents lead sulphate, and, multiplied by .6832, gives the weight of the lead. As calcium sulphate is soluble in ammonium acetate, great care must be taken to insure its absence from the first-weighed residue by thorough washing.

ANOTHER METHOD.—Treat the ore as before up to the point at which the filter with its contents is laid in the dish, then, instead of adding ammonia, etc.,

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\* If an ashless filter is used this will be unnecessary.

add a strong solution of sodium carbonate, and either digest hot for from half to three-quarters of an hour, or, better, let it stand two hours cold, in either case stirring at intervals with a glass rod. The assay must be kept alkaline, more soda being added if required. The effect is, to convert the lead sulphate formed by the acid treatment into carbonate.

Now transfer the substance to a new filter; pour a little water on the old filter still in the dish, and work it with a glass rod until it is reduced to a pulp, and all transferred to the new filter. Wash with hot water till the drips no longer cause turbidity in a little solution of barium nitrate or chloride in a test-tube. Place a clean beaker under the filter and pass warm, dilute nitric acid through the assay, which must be kept covered by a watch-glass with the concave side upward, or with a small inverted funnel, to prevent loss by effervescence.

Pour the acid in little by little, so that it may have time to dissolve the lead carbonate. Continue this until the drips no longer give a p. p. in dilute sulphuric acid, returning the tests to the filtrate.

If preferred, instead of dropping the nitric acid on the substance in the filter, the latter with its contents may be placed in a dish, there digested in the dilute nitric acid, and finally transferred to another filter and washed with hot water. In either case, to the filtrate add dilute sulphuric acid in liberal excess, and finish as in the first-described process. The lead sulphate may be reduced to metal for the weighing by fusion, at as low temperature as possible, in a porcelain crucible, with several times its weight of potassium

cyanide, and this is the safest way when ore containing strontium is treated by the sodium carbonate process.

If acetic acid instead of nitric acid is used to dissolve the lead carbonate, the drips may be tested and the lead finally precipitated as chromate, by solution of potassium dichromate, the p. p. collected, washed, dried on the water-bath and weighed on the tared or counterpoised filter. This method will insure the absence of lime. The lead chromate contains 64 per cent of lead.

The nitric solution may be converted into acetate for this determination by adding to it an excess of sodium acetate, that is, a little more than enough to saturate the nitric acid with sodium, so that the lead nitrate is converted into acetate.

Lime is the chief common constituent of lead ores which is likely to cause an erroneous result. If calcium sulphate (sulphate of lime) is allowed to remain with the lead sulphate at the first filtration, it will also be converted into carbonate by the sodium carbonate, or it, as well as the lead sulphate, will be dissolved by ammonium acetate. In either case it is liable to be thrown down again, together with the lead, as sulphate in the last precipitation. But while lead sulphate is *very* little soluble in water, especially if the water contains sulphuric acid, calcium sulphate is considerably so. It is for these reasons that stress is laid on the use of quite dilute acid for the precipitation, and of acid water for the washing.

The quantity of ore used in the wet assay may be from 10 grains or 1 gramme upward, according to the richness of the ore or the delicacy of the balance in use.

## COPPER ORES.

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**DRY ASSAY.**—The fire assay of copper is not very satisfactory, especially in unpractised hands; it may, however, answer the purpose of a prospector. The ore must be ground to pass a 40-mesh sieve. Poor ores, and all which contain lime or baryta and sulphur in any form, should be submitted to a matte smelting; the first in order to obtain an enriched product on which to operate; the second in order to remove calcium or barium sulphate which would cause a loss of copper. It is well to add a large nail to a matte smelting, to separate any lead which may be present.

ORES CONTAINING LESS THAN 8 PER CENT OF COPPER.

Ore.....	400	grains.
Borax glass....	400	"
Flour.....	20	"
Sulphur.....	10	"

Mix by grinding in wedgewood mortar; put in pot, cover with salt, and over that put 400 grains more borax glass. The sulphur and flour may be omitted if the ore contains sulphurets, zinc blende excepted, provided none of the higher metal-oxides are present. If there is any suspicion of lead, add a nail. Melt slowly and finish with strong heat. Remove the nail;

cook and break the pot. Separate the matte from the overlying slag and from lead, if any, and reserve for further treatment.

**RICHER ORES WITH LIME OR BARYTA GANGUE AND CONTAINING SULPHUR EITHER AS SULPHURETS OF ANY KIND, OR COMBINED WITH THE GANGUE AS GYPSUM OR HEAVY SPAR.\***

Ore.....100 grains.

Borax glass.....100 “

Sulphur if required.

Mix. Put in pot, cover with salt and more borax, and add a nail if lead is suspected. Melt, etc., as before. Reserve the matte.

**RICH SULPHURETTED ORES WITHOUT LEAD, LIME OR BARYTA.**—These include the mattes from the preceding as well as natural ores. Pulverize; roast in a roasting dish in the muffle, beginning with a low heat, and raising it gradually to bright redness, stirring the ore at intervals with a piece of wire. If the ore is inclined to decrepitate and fly out of the dish, it must be covered by another roasting dish inverted and having some notches in the edge and a hole in the middle, and must be cooled before being uncovered for the stirring. It will soon cease decrepitating. When no more odor is perceptible from the red-hot ore, cool, grind again with the addition of one-tenth part of coke. Roast again, observing whether arsenic is present or not. Arsenic is known by an odor like

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\* In this and preceding, strontia may be regarded in the same light as baryta.



that of garlic. Roast until all the coke is burned to ashes. If arsenic was observed, repeat the grinding with coke and roasting until it is all expelled. At last place on the ore a piece of ammonium carbonate of 10 or 20 grains weight, or mix that quantity in the form of powder, with the ore, cover and heat until the carbonate has evaporated; this removes any lingering sulphur. The ore is now in the following class.

**RICH OXIDIZED ORES.**—If the ore is a natural oxide, the assay must be weighed, and calcined by heating to redness in a roasting dish. Add to the assay—

Soda.....an equal weight,

Argol.....“

Lime.....one-fourth the weight,

and, if it contains much quartz, a little powdered iron-stone (hematite) or forge scale. Mix well in the wedge-wood mortar. Place in a pot which should not be more than two-thirds filled. Cover with a mixture of soda and argol in equal parts, and on that a weight of borax glass equal to the weight of the assay. Heat very slowly to dull redness for fifteen minutes, then fuse thoroughly with high heat, and remove from the fire without delay. Wash the side of the pot by the slag as in an assay for gold and silver, tap to settle all globules, allow to cool, and break the pot.

The copper should be found at the bottom of the pot in a single button. If the button is tolerably malleable, and when broken or cracked is found to be of a good red color all through, it is pure enough, and may be weighed. If it is black, gray, or mottled, it

must be refined by cupellation. The slag should be neither red nor green. The side of the pot should be but little colored with red or green.

**REFINING THE IMPURE COPPER.**—Place the button on a highly-heated cupel, and an equal weight of pure copper on another. Add to each one-tenth part of lead, and close the muffle after placing some glowing coals in it. When the buttons are well melted, open the muffle to allow the entrance of air; the refining then begins. The button behaves much in the same manner as one of silver, when nearly free from lead, but the final brightening is less vivid. The heat must be higher than for silver, and should be such as that the copper solidifies at the instant when the lead leaves it. The button should then appear greenish. If it does not seem to be pure, add another tenth of lead, both to the assay and to the pure copper beside it. Continue thus adding lead by tenths and cupelling it off, until the assay button is pure. Remove from the muffle and plunge both of the buttons into cold water as soon as possible after solidification in order to stop oxidation.\* Clean by hammering and brushing; weigh, and to the assay button weight add as much as the pure copper has lost. If pure copper is not at hand, refine the assay button and weigh it; then cupel it again with as many tenths of lead as were used in the refining; weigh again, and to the first weight add as much as has been lost in the second cupellation. The percentage must be calculated on

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\*Some operators cover the button with charcoal powder at the instant of brightening, with the same view.

the quantity of ore originally taken, whether matte smelted or not.

Argol is crude cream of tartar; an intimate mixture of one part of flour and four of soda may be used instead.

WET ASSAYS.—*Common Method*.—A suitable quantity of the finely-powdered ore is gently boiled with a mixture of nitric, sulphuric and hydrochloric acid, more of the first than of the last, until decomposed, the sulphur, if any be present, either being completely oxidized into sulphuric acid or melted to clear amber-colored drops.

It is best that the sulphur be oxidized; the oxidation is promoted by adding, from time to time, a few crystals of potassium chlorate. If the sulphur is not dissolved by oxidation, the assay must be cooled until the drops become solid so that they may be removed to a small dish and burned; any residue is then to be dissolved in hydrochloric acid and added to the main solution. Continue the boiling, adding a little nitric acid from time to time as long as it produces red fumes, and then boil until the odor of nitric acid is no longer perceptible and the dense white fumes of sulphuric acid are evolved, adding more sulphuric acid if necessary, so that the assay does not become dry. Cool, add water cautiously, pouring it under the slightly-lifted cover; rinse the cover into the vessel, keep warm for half an hour, stirring occasionally with a glass rod; filter and wash, receiving the liquid in a clean beaker. To the filtrate and washings in the beaker add some hydrochloric acid or ammonium

chloride, and then ammonia in excess, that is until the liquid smells strongly of it and a deep blue solution is formed.

If a considerable brown flocculent p. p. forms on the addition of ammonia, any arsenic which may be present will go with that; otherwise add "magnesia mixture" as long as it produces a turbidity. Let the assay stand as long as is convenient, in a warm place, then filter and wash with water containing ammonia as long as the drippings have a blue tint. To the filtrate add hydrochloric or sulphuric acid until it is quite acid; warm slightly, and place in the beaker a piece of clean sheet iron of such a size that, while completely covered by the liquid, it does not lie flat on the bottom of the vessel.

Heat the liquid gradually to almost boiling. Bubbles of hydrogen gas will be seen rising from the surface of the iron, and this action must be kept up, in moderation, by adding hydrochloric or sulphuric acid from time to time, if necessary. When the liquid is quite colorless, test it by holding a clean piece of iron in it for half a minute; if the iron is not discolored, no copper remains in the solution. A piece of zinc is even better than iron for the test, and pure zinc may be used in place of iron for the precipitation of the copper if it is certain that no nitric acid nor nitrates remained after the evaporation with sulphuric acid. The beaker should be covered during the precipitation of the copper, to exclude dust.

The copper is now in the metallic state, precipitated by the iron, a corresponding portion of which has been

dissolved. Pour off a great part of the liquid, with care that no copper goes with it. Transfer the remaining contents of the beaker to a dish, and detach the copper from the iron by means of a clipped feather. Reject the iron and wash the copper, first with cold water, then several times with hot pure water, draining it off as closely as possible each time into another dish in which any small particles of copper that may unavoidably escape may be settled and eventually recovered; the last two washings are best made with alcohol, but it is not essential; the spongy copper may be compacted by pressing with the finger, or the bottom of a test-tube, to aid the washing and draining. Some light black particles of carbon from the iron will probably be seen; these should be washed away as much as possible. A slight discoloration of the precipitated copper may often be removed by gentle boiling in dilute hydrochloric acid; the proper color is a pure copper red.

The washed and drained metal is to be dried in the dish on the water-bath. The percentage is calculated for the weight of ore used, which may vary, with the supposed richness and the delicacy of the balance, from 10 grains upward.

Russia sheet iron is suitable for the precipitation of copper if the glaze is removed by means of warm dilute hydrochloric or sulphuric acid and rubbing. Good wire cut to a suitable length is also proper; it may be cleaned with sand-paper; horseshoe nails are excellent. If zinc is used, it should be such as dissolves without residue in dilute hydrochloric acid.

Magnesia mixture, for the removal of arsenic in the case of an ore containing little or no iron, is prepared by dissolving epsom salts in water and adding, first sal ammoniac (ammonium chloride), and then ammonia; the ammonia should produce no p. p., and it will not if enough sal ammoniac be present.

The dried copper should retain a good color, and should not adhere to the dish in which it was dried, and it will not if well washed with pure water or alcohol; the result is more reliable if the dried copper is calcined at a red heat in the muffle, cooled, moistened with nitric acid, again heated to redness, cooled and weighed; it is then in the form of black oxide, of which 79.85 per cent is copper.

An oxidized ore, whether a natural product or the result of roasting a sulphuret, if free from arsenic, antimony, and bismuth, requires only to be decomposed by hydrochloric and sulphuric acid,\* the solution filtered and the copper precipitated by iron or zinc, etc.; the red oxide, however, requires an addition of potassium chlorate to the acid solution in quantity sufficient to convert that from a brown and muddy appearance to a clear green, after which it must be boiled for some time to expel excess of chlorine before the precipitation is attempted. Nitric acid will answer as well as potassium chlorate, only then the solution must be evaporated with sulphuric acid as in the first described process. Ores which do not yield all their copper to acids may be made to do so by previous fusion with soda in a porcelain crucible.

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\* If lead is present, boil down with sulphuric acid until all hydrochloric acid is expelled; also if there is much silver.

A better way in which to precipitate the copper is to pour the solution into a platinum dish of known weight, and add a piece of pure zinc; the copper is precipitated on the dish, to which it adheres, making the washing easy. The whole of the zinc is to be dissolved, acid being added, if necessary, for that purpose until no more bubbles are seen. The copper is washed, dried, and weighed still on the dish, the known weight of the latter deducted, leaving the net weight of the copper. The copper is removed from the dish by nitric acid, which does not attack the dish.

But little inferior is the method of precipitating the copper in a porcelain or glass vessel in which is placed a weighed piece of platinum foil, and, in contact with that, a piece of zinc; the copper is precipitated chiefly on the platinum. In these processes all nitric and hydrochloric acid is expelled from the solution by boiling down with sulphuric acid.

The method which is generally considered the best at present is the precipitation by electrolysis, in which a current of electricity is passed through the solution by means of platinum electrodes, on one of which the copper is deposited in a compact form. The nitric solution is used in this process, hydrochloric acid being injurious. The method requires special apparatus and experience, and though very exact, is equaled in this respect and surpassed in point of expedition by my new process, to be described shortly.

Ores containing selenium or tellurium should be roasted. In the case of ores containing metallic copper in pieces too large to pass the sieve, the process is

similar to that described for gold and silver ores containing coarse metal; (See Part I) if it is desired to avoid the calculation, the coarse metal may be dissolved in nitric acid, the solution dried and the residue calcined and mixed with the powdered ore.

*Aaron's Method.*—This assay is very accurate. The ore may contain all of the elements which are likely to be present in mixed ores of silver, lead and copper, except selenium and tellurium, which should be roasted off if present.

Digest a weighed quantity of the powdered and dried ore in nitro-hydrochloric acid and boil down with sulphuric acid until the fumes of that appear. Cool, dilute, and add a drop of hydrochloric acid to precipitate any silver which may have been dissolved from the chloride by the sulphuric acid.

Filter; to the filtrate add ammonia in considerable excess, and boil for five minutes; again filter, and, after slight washing, redissolve the p. p. in hydrochloric acid; again add ammonia, and filter into the original beaker, washing the p. p. thoroughly with ammoniated water. This double treatment is necessary to extract all the copper.

To the ammoniacal solution of copper thus obtained add hydrochloric acid in slight excess, then enough of sodium acetate to saturate all free hydrochloric acid, then some sodium sulphite in crystals, and finally several grammes of sodium thiosulphate, also in crystals. The liquid should become quite clear, and perfectly colorless unless tinted by chromium. Now add freshly dissolved potassium xanthate as long as it



precipitates, stir, to curdle the p. p., and collect that on a counterpoised filter. Wash with rather hot water, and then, if possible, with alcohol; dry on the water-bath and weigh on the filter.

The cuprous xanthate is of a fine yellow color; it contains 34.85 per cent of copper.

If the precipitate has a brownish color, cobalt is probably present, and the alcoholic washings will be tinged with green. Nickel is likely to be there also; the color of its xanthate is a reddish yellow, which does not show very plainly in the mixture unless in large proportion.

To free the cuprous xanthate from nickel and cobalt and at the same time recover those metals approximately, omit the washing with alcohol for the present and proceed as follows. Wash the mixed and still moist xanthates on the filter with slightly dilute ammonia until that passing through no longer gives a yellow or brownish-yellow p. p. on addition of acetic acid. The nickel xanthate may be recovered from the ammoniacal filtrate by neutralizing that with acetic acid and adding a little potassium xanthate. Having thus removed the nickel, again wash the substance remaining on the filter with a strong solution of potassium cyanide as long as that gives a yellow p. p. with acetic acid. The green cobalt xanthate remains on the filter.

To the filtrate add acetic acid in slight excess, collect the reprecipitated cuprous xanthate on a counterpoised filter, wash it free from potassium salts with hot water,

then displace the water by alcohol, if convenient, to facilitate drying on the water-bath.

The nickel and cobalt xanthates may be dried on the water-bath and weighed for approximate results. Any nickel xanthate which may have been left in the cuprous xanthate will have been dissolved by the potassium cyanide, but not reprecipitated by the acetic acid, hence will be lost. Owing to the high molecular weight of the cuprous xanthate only a small quantity of ore need be taken for the assay, one gramme being enough unless the ore is very poor, or a coarse balance is employed. The method is adapted to alloys also. If a red or dark p. p. or color appears on adding the sodium sulphite, selenium or tellurium is present and a new assay must be made. A white p. p. at this time indicates cadmium; it redissolves when the thiosulphate is added.

Cadmium is precipitated completely from an acetate solution (leaving zinc dissolved) by sodium sulphite, but not in presence of ammonia salts, hence the cadmium cannot be determined by this means in this assay.

*Assay by Amalgamation.*—Take 100 grains or more of the powdered ore, and, if it contains sulphurets, roast it. Grind it in an iron mortar, warmed, with water enough to make a pulp, and such a quantity of a mixture of iron sulphate and salt as may be necessary to cause a copper stain on a piece of clean iron held in the hot pulp for a few seconds; also a little mercury. Continue rubbing and stirring with the pestle until

the latter no longer takes up mercury when lifted out, and a bright piece of iron is not stained by the pulp, then wash the pulp away by a stream of water, leaving the mercury. Strain the mercury through a piece of wet buckskin by twisting. Copper, perhaps also lead, gold, and silver, will remain in the form of a ball of amalgam. Tie the ball in a small piece of cotton cloth, as a dumpling is tied; put it into a crucible; cover it with powdered charcoal; heat it gradually to redness; then add some borax and a piece of charcoal and heat strongly. When cold, break the pot, in which a button of metal will be found. If the button is good copper, weigh it; if not, refine it as directed for the crucible assay, and then weigh. If the button remains of a pale color after refining, it probably contains a large proportion of silver; treat it by scorification and cupellation for precious metal. From the net weight of copper obtained, calculate the percentage for the weight of ore taken.

**VOLUMETRIC METHODS.**—There are many methods of estimating copper volumetrically, that is, by the *volume* of a reagent which is found to be necessary to produce or complete a certain effect in the suitably-prepared solution; the one most commonly used depends on the fact that the blue ammoniacal solution of copper is discolored by a solution of potassium cyanide; this is generally known as the—

*Cyanide Process.*—Take 10 grains, or 1 gramme, of pure copper; dissolve it in nitric acid; add ammonia until the p. p. at first formed is redissolved to a blue

liquid; add water to make about one-third of a pint (in a beaker). Charge a Mohr burette with a solution of potassium cyanide made by dissolving one troy ounce in five and a half fluid ounces of water (or the water may be weighed) and filtering if necessary. Run the liquid from the burette into the blue solution of copper, stirring that with a glass rod, until the blue color becomes faint and purplish; continue cautiously, adding the cyanide little by little, finally drop by drop with intervals between, until only a faint tint remains, which disappears in a few minutes, leaving the liquid colorless; observe how many c. c. of the cyanide have been used, by the marks on the burette, and note the number. The cyanide solution is thus *titrated*, that is, it is known what volume of it corresponds to 10 grains or to 1 gramme of copper in the assay. The titrated cyanide solution should be kept in a dark-colored stoppered bottle.

To assay an ore, take 20 grains or 2 grammes, more or less according to the grade of richness, and make an acid solution by one of the methods previously described, or by nitric acid alone; it is not necessary to expel nitric acid in this process. Add ammonia in excess, and heat moderately for some time. If there is any considerable quantity of insoluble matter, filter and wash with water containing ammonia until that passes through colorless.\* The entire volume of solution, including the washings if filtered, should be about one-third of a pint. When quite cold add solu-

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\* For accurate work the p. p. should be redissolved, and again precipitated by ammonia, the new solution being added to the other.

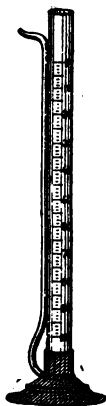
tion of potassium cyanide from the burette precisely as in titrating, noting the quantity used.

The determination of the assay is now a mere matter of proportion. For instance, if 10 grains of pure copper required 45 c. c. of cyanide solution, and the ore assay required 63.5 c. c., then  $45:10::63.5:$  the copper in the assay  $=14.1$  grains, and if 20 grains of ore were taken, the percentage is  $14.1 \times 5 = 70.5$ , or, if 50 grains of ore were used,  $14.1 \times 2 = 28.2$  per cent.

The presence of manganese interferes a little with the discoloration; this is obviated by an addition of ammonium carbonate. Arsenic also interferes, but may be prevented by "magnesia mixture," added before filtering. Nickel, cobalt, or zinc is fatal to the accuracy of the process, and, when either of these is present, the copper should be precipitated by iron or zinc in the first described process, washed, dissolved in nitric acid, blued by ammonia and determined as above.

The process is rapid and easy, therefore popular, but not always accurate. The quantity of ammonia and of the different acids used affects the result and should therefore be, as nearly as possible, the same in the assay as in the check on pure copper, which should always be made with each set of assays of ore or alloy.

The cyanide solution is liable to change, hence too large a quantity should not be made at one time; the directions given above for making it only indicate approximately the proportions to be used, which may be modified to suit convenience, and the quantity prepared may be adapted to the expected demand.



Gay Lussac  
Burette.

The cuts show two kinds of burettes which are suitable for this and other volumetric assays. The Mohr burette is the most convenient; it may be of 50 or 100 c. c. capacity, should be graduated to one-tenth c. c. and furnished with Erdman's float. The discharge by means of rubber tube and pinch-cock is preferable, for common use, to that by a glass cock.

*Aaron's Method.*—Copper in ammoniacal solution is completely precipitated by solution of potassium xanthate. By this reagent copper is easily detected in a solution which is not visibly blued by addition of ammonia. Neither arsenic, nickel, zinc, manganese, nor the earths interfere. So delicate is this method that it is possible to make the assay on one decigramme of ore to one-tenth of one per cent, but I prefer to operate on larger quantities when accuracy is requisite.



Mohr  
Burette.

Weigh ten grains, or one-half gramme, of pure copper, and dissolve it in nitric acid; add ammonia in excess, and water to make about half a pint of solution, in a beaker of thin clear glass; from a Mohr burette, which should be graduated at least to one-fifth c. c., run in a solution of potassium xanthate, stirring gently the while, or at short intervals, with a glass rod. The solution in the beaker soon appears green from admixture with the yellow p. p. As long as a green

color is plainly seen on stopping the flow of the xanthate, and gently stirring, more xanthate may be freely added; when the green color grows faint, proceed with caution; stir actively for half a minute or more, but avoid making froth; allow the p. p. to settle to some extent, and if the liquid is not clear enough stir again, but as long as a green tint is perceptible it need not be very clear. Continue adding xanthate, by quarter c. c. or less, stirring and settling. When near the end, stir and settle until the liquid is no longer turbid, although flecks of the p. p. may remain suspended; after this a very little stirring will suffice after each addition of a drop or two of xanthate. When an addition of xanthate causes no cloud in the liquid, the precipitation is complete and a slight excess of the xanthate is present.

For the purpose of verification, a second assay may be made, and nearly the quantity of xanthate required run in at once, finishing with caution as before. To verify the presence of a slight excess of xanthate, stir and settle, then draw through the clear liquid the end of a small glass rod moistened with an ammoniacal solution of copper; either a faint cloud will appear near the surface, or minute drops of the blue solution will sink through the liquid, leaving thin white streaks behind them.

Toward the end of the operation, a record should be kept of each quarter or tenth c. c. of xanthate added, in order that, in finally reading the burette, the last addition, which produced no p. p. and was therefore not required, and half of the one before the last,

because it was probably only partially requisite, may be rejected; thus it is known how many c. c. of the xanthate are required to precipitate the quantity of copper taken, which constitutes the *titre* of the solution (or the *standard*).

To assay an ore, take such a weight as may be supposed to contain from one to ten grains of copper, or a corresponding proportion in grammes if that system of weight is in use. Treat with a mixture of nitric, hydrochloric and sulphuric acid, boiling gently in a covered vessel or a matrass, until decomposed and all sulphur is oxidized;\* cool, dilute, filter and wash. To the filtrate and washings add ammonia in excess; filter, wash with water containing ammonia until it gives no yellow color on testing a little of it in a test-tube with a glass rod moistened with xanthate solution. Let the entire volume of solution be about half a pint, adding water if necessary to complete that quantity, then proceed with the xanthate as before. From the number of c. c. used, and the titre of the xanthate, calculate the quantity of copper in the assay, as in other volumetric methods, and from this quantity reckon the percentage on the weight of ore taken.

In making this assay, the xanthate should at first be run down the side of the beaker, to prevent frothing and consequent floating of the p. p.; the smaller quantities added toward the last are best allowed to fall upon the stirring rod resting in the beaker. As long as much p. p. is formed, it may be plainly seen

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\* If silver is present, boil with sulphuric acid until the fumes of that come off.



by looking downward on the solution, but the faint clouds produced later are best seen, as in the humid assay of silver, by holding the glass toward the light, and looking obliquely upward through the liquid. The liquid clears better if there is a goodly quantity of p. p. than otherwise; it clears best when the precipitation is near completion. Excessive stirring is disadvantageous by breaking the curd; hence stir but little, and gently, until a more perfect clearing becomes necessary, then agitate briskly for a minute or two, and settle enough to leave a stratum of clear liquid at the top before adding more xanthate, after this a very slight stirring suffices.\*

There are three guides to the ending: Firstly, the disappearance of the green color, and darkening of the yellow; secondly, the more perfect clearing, and thirdly, the cessation of clouding on addition of xanthate. To make the assay well some practise is required; the novice is almost sure to run in too much of the xanthate. It would be quite feasible to have a titrated solution of copper with which to work back by adding a known quantity, and, in the final reckoning, deducting a corresponding weight from the total result; the most convenient way in which to make such a solution is to dissolve some bluestone in water, and then titrate a certain measure of the liquid, which should be quite a weak solution; to use it, measure the required quantity, add ammonia, and pour the whole into the assay; then proceed again with xanthate.

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\* The presence of ammonium chloride promotes curdling and settling.

In order to be quite sure in the case of a very complex ore, boil the first acid solution, before filtering, until heavy white fumes of sulphuric acid come off; cool; add a little water and a piece of iron; keep the liquid acid with sulphuric acid, and digest warm until the liquid no longer stains a piece of clear iron, filter, wash, redissolve the precipitated metal in mixed acids as before; add ammonia, filter and proceed with the assay.\* This will be necessary when an appreciable quantity of cobalt is present in the ore. The copper p. p. should be orange yellow, although it may appear darker at first; if it is distinctly brown there is probably cobalt in the assay, or the xanthate may be out of order, which can be tested in a moment by means of some ammoniacal solution of bluestone.

After the precipitation of the copper, a slight excess of xanthate being present, if acetic acid, added to the filtered liquid in slight excess, produces a reddish yellow p. p., nickel is present; and if greenish turbidity, either at once or on further addition of xanthate, cobalt is also present, and another portion of it will be with the copper p. p., spoiling the assay, which must then be repeated as directed above.

PREPARATION OF THE POTASSIUM XANTHATE SOLUTION.—Dissolve two ounces (troy) of caustic potassa in about a quarter pint of 95 per cent alcohol in a stoppered bottle; add carbon disulphide, little by little, as long as a curd is produced, a hissing noise heard, and a partial vacuum is formed in the bottle, as evinced by the stopper resisting extraction. Shake the bottle oc-

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\* If silver is present, filter before adding ammonia and again after.

casionally. When the above effects cease, and a further addition of the disulphide causes expansion, which may expel the stopper, while its odor is no longer neutralized but becomes disagreeably perceptible, and a yellowish fluid appears which partly liquefies the previously almost solidified contents of the bottle, pour the whole into a dish and leave it to spontaneous evaporation in a dark, cool place for twelve hours or more, the dish will then contain a plastic mass; dissolve this in six pints of cold, pure water. Let the solution stand a short time to deposit any excess of disulphide, and then bottle it in common beer bottles ("black bottles"), which keep in a cool, dark place.

About 50 c. c. of the solution thus made will precipitate 0.5 gramme of copper; it may be diluted, if so preferred for very close work, so that it will require 100 or 200 c. c. for that quantity of copper. If the alcohol is not almost saturated with caustic potassa, the curdy mass spoken of may not appear until evaporation has taken place. The compound may be made in an open dish, the carbon disulphide being dropped into the alcoholic solution of potassa until that no longer reacts alkaline. The crystals of potassium xanthate are white; they may be washed with ether, and may be preserved indefinitely, and dissolved in water or in alcohol for use. Caustic soda may be used in place of potassa; the sodium xanthate forms orange-colored crystals.

## TIN ORES.

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TIN stone (tin oxide) is readily smelted to a button.  
Take—

Powdered ore . . . . .	1 part
Potassium Cyanide . . . .	$\frac{1}{2}$ “
Soda . . . . .	$\frac{1}{2}$ “

Mix. Put in pot; heat quickly. When fused, leave five minutes in fire, then shake, tap, allow to cool, and break the pot. The button is silver white, malleable; a piece of the metal boiled in nitric acid forms a white powder insoluble in water. Antimony, which can be smelted in the same way, acts similarly in nitric acid, but antimony is neither white nor malleable; it is gray, crystalline, and brittle. (See Part I for distinction.)

The presence of quartz (silica) causes a large loss of tin in the smelting, hence, if the ore is not pure, it should be washed by “panning,” or vanning, in order to remove the quartz as far as possible from the heavier tin oxide before smelting.

Mixed ores containing tin, or very impure tin ores, should be boiled, in the state of fine powder, in a mixture of three parts hydrochloric and one part nitric acid.\*

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\* From Mitchell.

Heat gently for half an hour, then boil until the greater part of the acid has evaporated. Cool, add water, settle, and pour the water off, not losing any solid matter. Wash thus until the water is tasteless; the residue is tin oxide, quartz, and tungstic acid if the ore contained tungsten, as it often does. The tungstic acid is removed by gentle heating for an hour with ammonia, stirring occasionally; the ammonia is then poured off and the residue washed. The quartz is removed by vanning, and the tin oxide smelted as before.

## MERCURY ORES.

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THE principal ore of mercury is the red cinnabar for which red iron-oxide is so often mistaken; there are also black ores of mercury, and many silver, copper, and other ores contain small quantities of mercury.

Before assaying a doubtful ore for mercury it is well to test it. Boil some of the powder in a mixture of nitric and hydrochloric acid; dilute and place a drop of the liquid on a piece of gold; touch the gold, through the drop, by a pointed iron or steel implement, such as a penknife or a needle; if the ore contained a trace of mercury the gold will be instantly amalgamated on that spot, which will be whitened. Or, take a glass tube about six inches long and a quarter inch diameter, closed at one end; place in the tube about a grain of the ore mixed with two or three grains of litharge. In order to get the charge to the bottom or closed end of the tube without soiling the other part, line the tube with a rolled slip of writing paper (or glazed paper), introduce the charge and tap it down, then remove the paper. Heat the end of the tube containing the charge in the flame of a spirit lamp, or a candle. If mercury is present, it will first evaporate, and then condense in the cool part of the tube, in the form of minute globules which appear like a mist if

the quantity is very small; cut the tube a little by a file near the charge, and break off the part containing the mercury. By means of a wire and a bit of moist cotton cloth, wipe the interior of the tube, the globules of mercury will be visible on the swab, by the aid of a magnifying glass, or may be washed off in a cup of water. By weighing the ore, using a longer tube kept cool by a moist cloth toward the open end, and carefully collecting and weighing the mercury, the operation becomes an assay.

*The assay—*

Ore.....	1 part
Lime.....	$\frac{1}{3}$ "
Charcoal.....	$\frac{1}{10}$ "

Mix. Put into an iron or clay retort; cover with lime and charcoal; close the retort and heat to bright redness, keeping the retort pipe cooled by a stream of water flowing on a cloth wrapped around it; receive the condensed mercury in a vessel of water placed under the end of the pipe. The pipe should not dip into the water, but a piece of cloth tied around it may do so. Iron filings or borings will answer in place of lime; use one part to two of ore for the mixture, and some more as a cover. Any mercury which may remain in the pipe must be washed out. Pour the water off from the mercury; dry that by means of filter paper, weigh, and report in percentage of the weight of ore taken.

A small assay may be made in a tobacco pipe used as a retort, being closed by means of clay. A small amalgam retort is suitable. A quicksilver flask with

a pipe adapted will answer for several pounds of ore; the flask should be previously heated to redness to drive out any quicksilver which may be adhering to it, owing to its having contained quicksilver before. Gas-pipe alone may be employed; a short piece, plugged at one end, serves to receive the charge, and this is connected by a sleeve with a longer piece. It is better if the short piece is of the greater diameter, say two inches, and connected by a reducer with a longer piece of  $\frac{3}{4}$ -inch pipe. When the distillation is finished, the pieces are disconnected and the longer one washed for recovery of any adhering mercury.



## ZINC ORES.

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**GENERAL METHOD.**—The ore may contain, besides zinc, indium, iron, manganese, nickel, cobalt, copper, silver, lead, mercury, bismuth, antimony, arsenic, tellurium, selenium, tin, molybdenum, sulphur, phosphorus, silica, and the earths.

Take from 10 to 50 grains, or 1 to 5 grammes. Decompose by boiling in a mixture of sulphuric, nitric, and hydrochloric acid; add some water and then excess of ammonia, also some ammonium carbonate. Keep warm for some time; filter, wash with ammoniated water till the drips give no p. p., dark or white with ammonium sulphide. To the clear filtrate (and washings) add ammonium sulphide\* as long as it precipitates and until the liquid smells of it. Stir, settle, filter, keeping the filter as nearly full as may be, and covered as much as possible.

Wash with water containing ammonium sulphide, keeping the filter covered as before. When finally drained, add a drop of ammonium sulphide to prevent oxidation of the p. p. and consequent loss of zinc. If the p. p. consists only of zinc sulphide, it will be white, otherwise, as in presence of silver, copper, nickel, or cobalt, it will be mixed with black, while a yellow p. p.

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\*The ammonium sulphide should be free from carbonate, and saturated with hydrogen sulphide.

shows that cadmium is present or some arsenic remained, after the treatment with ammonia, which might have been removed by an addition of magnesia mixture; these substances may be disregarded. Manganese sulphide, being of a pale flesh color, is not conspicuous in presence of zinc sulphide; however, if present, it may be removed by washing the p. p. with acetic acid, and afterward displacing that by water.

Place a clean beaker under the filter and, not entirely uncovering the latter, pour on the edge, all round, some warm, rather dilute, hydrochloric acid; this will dissolve the zinc sulphide, leaving copper, nickel, cobalt, cadmium and arsenic sulphides unacted on.

When all the zinc seems to be dissolved, again place a clean beaker under and pour the solution so far got again into the filter. When the liquid has passed, pour some more of the dilute acid on the edges and afterwards wash with water. The zinc is now in the filtrate.\*

You have a choice of methods for the determination of the zinc; 1st, as zinc oxide; 2nd, as ammonium-zinc phosphate; 3d, volumetrically.

*As zinc oxide with 80.26 per cent of zinc.*—Heat the solution to boiling and add a crystal or two of potassium chlorate. Continue the boiling, with addition of small quantities of the chlorate, until any turbidity which may appear clears up and a smell of chlorine is perceptible; it may be necessary to add a few drops of

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\* In case there should be much sulphide of copper, etc., it will be better to transfer the p. p. to a beaker and there digest it in warm dilute hydrochloric acid, then filter and wash.

hydrochloric or sulphuric acid, in case not enough was present. To the clear solution, in a large covered beaker, add solution of sodium carbonate as long as it precipitates; heat to boiling for 20 minutes; collect the p. p. on a filter; wash until the drips no longer trouble a solution of silver nitrate and are neutral to test paper; dry, transfer the p. p. to a porcelain dish, add the ash of the burned filter; \* calcine at a red heat as long as the weight diminishes (cool and weigh from time to time). The zinc is now in the form of oxide, yellow while hot, white when cold.

*As zinc-ammonium phosphate with 36.49 per cent of zinc.*—After boiling with potassium chlorate as above, cool somewhat and add ammonia in sufficient excess to redissolve any p. p. that may at first appear, or otherwise until the liquid smells strongly of it; add some ammonium carbonate. If there is any p. p. which will not redissolve in excess of ammonia, filter; wash with ammoniated water.

Again add hydrochloric acid to the clear liquid, cautiously while stirring, at last quite dilute and drop by drop until the liquid is *very* slightly acid, so as to impart a purple tint to blue litmus paper. Add solution of sodium phosphate as long as it precipitates, and a slight excess; heat nearly to boiling until the p. p. settles; collect on a counterpoised filter; wash until

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\* The filter should be well cleaned, and, in order to avoid loss of zinc while burning it, a good plan is to wet it with solution of ammonium nitrate, then dry and set fire to it; thus any zinc carbonate which it may retain will not be reduced and volatilized. The ammonium nitrate leaves no residue to cause an erroneous result as ordinary nitre would.

the drips no longer trouble silver nitrate solution ; dry on the water-bath ; weigh on the filter.

In preparing the solution, if there is a large p. p. of iron, etc., on the first addition of ammonia, it should be redissolved in hydrochloric acid, after slight washing on the filter, and again precipitated by ammonia, etc., as before, the resulting filtrate being added to that first gotten. If chromium is present, as, for instance, in an ore carrying lead chromate, add solution of barium chloride to the first acid solution as long as it precipitates, and then stir in a pulp of moistened barium carbonate as long as it dissolves, and a little more. Let the assay remain cold, for six hours or longer, with frequent stirring. The iron, chromium, indium, and aluminum will go down. Filter; to the filtrate add ammonia, etc., as above. Or chromium may be removed by boiling the first ammoniacal solution, before filtering, for ten minutes ; filter hot, and wash at first with hot, slightly dilute ammonia, then with water. For description of volumetric methods, see other works. The volumetric processes are good where zinc assays are frequent.

AARON'S METHOD.—The ore may contain, besides zinc, the following elements in combination with each other or with oxygen, sulphur, or phosphorus, namely: Iron, manganese, copper, bismuth, silver, lead, mercury, arsenic, antimony, tellurium, tin, chromium, aluminum, magnesium, calcium, barium, strontium, silica ; to which may probably be added selenium.

Having weighed a suitable quantity of the powdered and dried ore, proceed as follows ;—

Decompose by boiling in a mixture of nitric, hydrochloric and sulphuric acid; let the nitric be in excess of the hydrochloric, and boil down with sulphuric until the heavy fume is seen and but little acid remains. Add water, carefully on account of spirting; again boil, and after the lapse of a few minutes to allow the soluble sulphates to dissolve, and still boiling gently, add solution of sodium thiosulphate (commonly called hyposulphite) as long as it makes a dark p. p.; this throws the copper down. If an orange yellow p. p. also appears, disregard it; it is caused by the precipitation of a part of the arsenic. Lead, silver, antimony, tin, barium and strontium will have been rendered insoluble by the acids.

Filter and wash. Heat the liquid to boiling, and add crystals of potassium chlorate until an odor of chlorine is perceptible and any turbidity has disappeared (it may be necessary to add a few drops of sulphuric or hydrochloric acid). To the clear liquid add ammonia in great excess and a few grains of ammonium carbonate, then, after a few minutes, solution of sodium phosphate by drops as long as it seems to increase the turbidity, and a drop or two more. The ammonia precipitates iron, alumina, and mercury; the carbonate throws down any alumina and lime that may have remained dissolved, also a portion of manganese if there is much of that; the sodium phosphate completes the separation of manganese and magnesia. We have now to get rid of chromium; this is done by boiling for ten minutes, after which filter, and wash slightly. Redissolve the residue by pouring warm

dilute sulphuric or hydrochloric acid upon it in the filter, receiving the solution in the same beaker from which the liquid was previously filtered. Treat this solution exactly as before, with ammonia, etc., also boil it for ten minutes. Filter while hot, through the same filter and into the same vessel as before, and wash thoroughly, first with hot and slightly diluted ammonia, then with water. To the filtrate add a few drops of sodium phosphate solution, and let it stand a short time; this is to insure the complete removal of manganese, magnesia, etc., so that, if a p. p. forms, you must filter again. To the clear liquid add sulphuric or hydrochloric acid, which may at first be strong, but later must be very dilute, stirring, meanwhile, until the reaction is *faintly* acid; if, as is probable, a slight excess of sodium phosphate has been added, a white p. p. will form when the neutral point is approached; this is the zinc which begins to come down, and it is a useful indication of approximate neutrality. As soon as blue litmus paper acquires a purple tint when dipped in the liquid, add sodium phosphate solution as long as it precipitates, and a slight excess. Heat nearly to boiling until the p. p. curdles and settles, leaving the liquid clear. Filter on a counterpoised filter, wash, dry, and weigh. The net weight of the p. p. multiplied by .3649 gives the weight of the zinc.

Before washing the p. p. it is best to make sure that all the zinc is down; add to the filtrate a few drops of sodium phosphate; if that produces no cloud, divide the liquid in two parts; to the one part add a drop of dilute acid, to the other a drop or two of dilute ammonia; if a cloud is produced by either test, the pre-

cipitation is imperfect and must be completed and the liquid again passed through the filter. In my experiments with this process I got from 98 to 99 per cent of the zinc actually present. It will be observed that no sulphides are used as precipitants.

In the absence of copper, the treatment with sodium thiosulphate may be omitted, as also then the boiling with potassium chlorate. If there is no chromium, it is not necessary to boil the ammoniacal solution, though it may be as well to heat it.

The advantages of the process are—

1st. Rapidity of execution, especially if copper and chromium are absent.

2nd. The use of none but stable reagents and the avoidance of troublesome sulphides.

3d. The high molecular weight of the product.

4th. Avoidance of calcination and of the loss of zinc which must be caused by the burning of the filter.

I believe it has the defect of yielding any cadmium that may be present as though it were zinc, and cadmium is commonly associated with zinc in ores. But it is very rarely the case that the cadmium is in sufficient quantity to cause an important error; when it is so, the first described or general method of assay may be used. Cadmium may be detected, either in the ore or in the final precipitate of zinc, by mixing a little of the substance with sodium carbonate and heating the mixture on charcoal before the blowpipe, when, if a notable quantity of the metal is present, it will form a brown coat on the coal, which is seen best when cold. The heating should not be too much prolonged, nor too high, or zinc will also coat the coal.

## AARON'S ASSAY OF NICKEL AND COBALT.

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NICKEL and cobalt usually occur together. The substance may contain, besides these metals, iron, manganese, zinc, copper, gold, silver, lead, bismuth, antimony, tin, arsenic, calcium, barium, strontium, aluminum, magnesium, silica, and sulphur. Should it also contain mercury, tellurium, or selenium, those are easily expelled by roasting of the weighed assay before proceeding further.

Powder very finely, unless the substance is an alloy, when it may be cut or rasped; take ten grains or one gramme, or less if the substance be rich; digest in a plenty of nitrohydrochloric acid, the nitric in excess. Let all sulphur be dissolved, or remove the drops from the sufficiently cooled liquid, burn them in a small basin, dissolve the residue, and add it to the main solution. Add a little sulphuric acid, unless the assay contained a good deal of sulphur, as copper or iron pyrites, evaporate nearly to dryness; cool, dilute, filter, wash with hot water until the drippings no longer give a dark p. p. with ammonium sulphide. (Test a small drop on white porcelain.) If the volume of liquid is now conveniently large, reduce it by evaporation. Add solution of caustic potassa or soda very carefully,



not producing any distinct p.p., although in many cases a slight turbidity, but the liquid must remain slightly acid to test paper.

If too much alkali be added, it must be counteracted by a drop or two of hydrochloric acid; it is better to use no alkali than too much.

Now add moist precipitated zinc carbonate; stir, and continue adding the carbonate until a considerable excess of it remains unchanged in the liquid, which may be slightly warmed. If the assay contained no copper, test a minute drop on white porcelain, with a drop of solution of potassium sulphocyanate; when this gives no red or pink tinge, the iron is all down. If copper is present, test for that, in the same way, with potassium ferrocyanide, which will give a brownish red color as long as any copper remains in solution; as the iron goes down first, it is not necessary to test for that when copper is present; when the copper is all, or nearly all down, a bluish or greenish p.p. may be produced by this test, which must not be mistaken for the blue which a persalt of iron would produce; it is caused by cobalt or manganese.

When the copper is all down, filter, and wash with hot water as long as the drips give a p.p. with solution of potassium xanthate. (Return these tests to the filtrate.) For a final test of the washings, place a drop on porcelain and add a drop of ammonium sulphide; it should not blacken. Now warm the liquid to about blood heat, or a little higher, and add solution of potassium xanthate; stir, and continue adding the xanthate, at intervals, as long as it is seen to produce a

yellow or a greenish p. p. Keep the liquid warm, and let it stand some time, stirring occasionally, until it clears tolerably. Try a little more xanthate; it will probably make a white p. p. of zinc xanthate, which, however, either changes its color or disappears, being either converted to nickel or cobalt xanthate, or dissolved. At last, filter a little of the solution on a filter which has been exactly balanced with another on a fine balance. Test the filtered liquid with potassium xanthate; if any yellow or green p. p. forms, return the test to the main solution, and add more xanthate to that, but avoid a great excess.

When no more p. p. forms, or only a white one which dissolves, make a final test of a drop with ammonium sulphide; it should give no black p. p., although a white one of zinc, perhaps tinged by pinkish manganese sulphide. Collect the p. p. after letting it settle on the same filter that was used in testing; any p. p. which may adhere to the side of the beaker must be removed by the aid of hot water and a rubber-tipped glass rod; a short piece of pure rubber tube on the end of the rod will answer. Wash several times with rather hot water.

Place a clean beaker under the filter, and leach the p. p. with slightly diluted ammonia until that passes colorless, and a drop of it gives no p. p. with a drop of acetic acid (or *very* dilute sulphuric) enough to destroy the smell of the ammonia. Add the drop to the filtrate if it gives a p. p.

Remove the beaker containing the ammoniacal solution of nickel xanthate, and wash the cobalt xanthate

on the filter with hot water containing a little ammonia, until the filter is clean and the p. p. separates well from it under the jet. Dry the cobalt xanthate, still on the filter, and the counterpoise filter with it, on the water-bath; cool and weigh it, on the filter, putting the counterpoise filter on the weight pan; return both to the water-bath for ten minutes; cool and weigh again, and so until it weighs twice alike.

The cobalt xanthate should be of a dark, pure, but not brilliant green color; if mixed with orange yellow, it contains a considerable proportion of copper; when calcined, it should be black, not green, which indicates the presence of zinc, and if it gives a blue solution with ammonia, copper is present; the copper may be removed thus: Redissolve the calcined p. p. and the burned filter, in aqua regia; dry with addition of hydrochloric or sulphuric acid; take up with water and a little of the same acid; digest warm with a piece of zinc, keeping the liquid distinctly acid so that bubbles rise from the zinc;\* the copper will be precipitated; remove the zinc and copper, nearly neutralize the liquid by potassa, reprecipitate the cobalt by potassium xanthate, wash, dry and weigh.

To the ammoniacal solution of nickel xanthate, add *very* dilute sulphuric acid until the greater part of the nickel xanthate is reprecipitated, but the liquid still smells of ammonia; then add acetic acid until the liquid has a slight acid reaction; stir, to curdle the p. p.; let stand for some time, stirring occasionally; add a little

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\* If the solution is allowed to become neutral or very nearly so, some cobalt will be precipitated on the zinc.

potassium xanthate, and, if it produces a p. p., add as much more as may be necessary, but avoid great excess. Collect on a counterpoised filter, cleaning the beaker as with the cobalt, wash with hot water until the p. p. cleaves readily from the filter under the jet; dry on the water-bath, cool, weigh, and so on until it weighs twice alike.

Each of the xanthates contains 19.6 per cent of metal, or, the weight multiplied by .196 is the weight of the nickel or cobalt. The filtrations may be greatly facilitated by connecting a foot of rubber tubing to the beak of the funnel and letting it hang downward, the end in the receiving vessel. This should not be done while leaching with ammonia.

In certain circumstances, believed to be the presence of arsenic in the solution, the precipitation of copper by zinc carbonate is not quite complete; if, however, there is sufficient iron present, the difficulty no longer exists, hence, if a little copper should remain in solution, notwithstanding an excess of zinc carbonate, add a little iron perchloride, as nearly neutral as may be, and then more zinc carbonate if necessary. If the ore is known to contain arsenic, with but little iron, while copper is also present, it may save trouble to add a little metallic iron to the assay in the first instance. All iron in the solution must be in the form of a persalt, and it will be so if enough of nitric acid be used in making the solution. In the treatment with zinc carbonate, the liquid may be slightly warmed, not highly heated, or nickel and cobalt will also come down.

The potassium xanthate made, as directed for the

copper assay, with alcohol of 95 per cent or thereabout, is a little alkaline, and becomes more so by keeping; in this state it is not fit for this assay. Before using it, test it with faintly reddened litmus paper; if it blues that, add some very dilute sulphuric or hydrochloric acid, not strong enough to cause turbidity, or, if it does so, warm the liquid until clear.

To make the zinc carbonate, or rather hydrocarbonate, dissolve zinc sulphate in water, and filter it; also dissolve sodium or potassium carbonate in water. Filter the latter into the former until nearly but not quite all the zinc is thrown down; collect the p. p. on a filter and wash it. The filtrate should still give a p. p. on addition of the soda solution, in order to guard against an excess of soda which would be difficult to wash out of the zinc carbonate. Keep the carbonate moist in a wide-mouthed jar or bottle. Probably zinc oxide would answer as well, and would be more convenient, because it would not cause effervescence.

*Resumé of Operations.*

1. Dissolve; dry; take up; filter.\*
2. Treat with zinc carbonate; filter.
3. Precipitate with potassium xanthate; filter.
4. Leach with ammonia.
5. Dry and weigh cobalt xanthate.
6. Recover nickel xanthate by acid; filter, wash, dry, weigh.
7. Multiply weights by .196 for metal. If the greatest possible accuracy is required, see *Fresenius' Quant.*, or send the assay to an analytical chemist.

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\* This filtration may probably be omitted without injury.

Sometimes a little of the cobalt is found mixed with the recovered nickel xanthate. To insure a perfect separation, collect the recovered nickel xanthate on the original filter, dissolve it again in ammonia and again recover it by acid as before, then collect it on a new counterpoise filter, dry and weigh. The whole of the cobalt will be on the first filter.

## ASSAY OF CHROMIUM.

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THESE methods are not for every substance which may contain chromium, but for chrome ore proper. Powder as finely as possible, take 5 grains or half a gramme, fuse in a platinum crucible with two parts of nitre and 4 of dried sodium carbonate; add the soda, etc., a little at a time, and stir with a stout platinum wire. Cool, and dissolve in hot water (put crucible and all into the dish), filter; acidulate with sulphuric acid, add ammonia in excess and let stand, hot, for an hour; filter; add hydrochloric acid in excess, and then solution of sodium sulphite; keep hot until the liquid is emerald green and the smell of sulphurous acid ceases. Alcohol may be used in place of sodium sulphite, but it acts more slowly. Precipitate by ammonia, and boil; filter while hot, and wash with hot water; dry the p. p., burn the filter, and add the ashes to the p. p.; ignite the whole; weigh, and deduct the weight of the ashes of a similar filter; report the percentage of chromium sesquioxide, which is what the p. p. consists of. The stirring with platinum wire during the fusion often prevents the need of a second fusion, but if any ore remains insoluble that portion must be collected, dried, and fused with a fresh quantity of soda, etc.

The platinum crucible must not come in contact with the fuel, therefore put it inside of a common assay crucible and pack lime around it, or operate in the muffle. A porcelain crucible may be used in case of need.

ANOTHER METHOD.—Fuse with nitre and soda, and dissolve in water as before. Filter; acidulate with acetic acid and boil to drive off carbonic acid. Dilute and add solution of neutral silver nitrate as long as a p. p. is formed. Collect the p. p. on a filter, and wash with pure water until the drips no longer give a p. p. with weak solution of salt, or dilute hydrochloric acid.

Boil the filter and p. p. in very dilute hydrochloric acid; cool, filter; wash and dry. The silver chromate first obtained is thus converted to chloride, which remains on the filter together with the previously-used filter. Place the whole in an assay crucible with some soda, borax, and litharge. Smelt; cupel the resulting button. The weight of silver got multiplied by 0.71 gives the weight of chromium sesquioxide in the assay.



## ASSAY OF BISMUTH.

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SMELT a suitable quantity of the powdered ore in an assay crucible, with borax and a mixture of equal parts of soda and potassium cyanide. Copper may be kept out of the button almost entirely by means of sulphur or arsenic, hence, if the ore contains copper, and but little arsenic or sulphur, or none, add sulphur and powdered charcoal in the dressing, also a couple of nails. In this way the bismuth can be got tolerably pure from very complex ores containing copper, zinc, nickel, cobalt, and tellurium. Gold and silver, if present, will go into the button, which should therefore be cupelled after weighing, and the weight of precious metal found deducted.

Lead and antimony, if present, will go into the button. With very poor ores, it is even advisable to add some litharge in the dressing, in order that the resulting lead may collect the bismuth; about 4 times the weight of the bismuth will suffice. In these cases, or if much copper has been allowed to go into the button, that must be purified. Laminate or powder it, according as it is tough or brittle, and digest it in boiling nitric acid, which must be considerably diluted if lead is present; this will dissolve the bismuth, lead, copper, and silver, leaving antimony (and tin) insoluble.

Add enough sulphuric acid to precipitate the lead; boil off some of the acid; cool, add water and boil. If there is any insoluble matter, settle; decant upon a filter; boil again two or three times with water and a little sulphuric acid, also a drop of nitric acid, decanting each time upon the filter. Finally throw the residue on the filter and wash it slightly; it should be tested by blowpipe for bismuth, which should not be found.

To the filtrate, in a large, covered beaker, add excess of ammonium carbonate, heat to boiling, filter, wash with water containing ammonia until that passes colorless, dry and remove the p. p. from the filter, which burn. Smelt the p. p. and the filter ash together, with a little borax glass and potassium cyanide, in a small crucible, cool and break the pot; separate the button from the slag with care; it may be cleaned by means of warm water with a little sulphuric or hydrochloric acid added.

## ASSAY OF ARSENIC.

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In a porcelain crucible fuse half a gramme (or less if rich) of the powder with 6 to 10 parts of a mixture of sodium carbonate and nitre (in equal parts). Cool; extract with water warmed in the crucible; filter. The filtrate contains the arsenic combined with alkali. Acidify with nitric acid, and boil. Cool; neutralize almost exactly by ammonia, so that previously reddened litmus shows alkali in half a minute, if not clear, filter. Add solution of neutral silver nitrate (lunar caustic) in slight excess. Stir to coagulate the p. p., which is of a brick red color. Filter; wash the p. p. with cold water. Test the filtrate with silver nitrate, dilute nitric acid and ammonia (to again neutralize) in order to see that precipitation is complete. Scorify the p. p. with lead, and cupel the button.

If the ore contained any chlorides, as silver chloride etc., or if the water or soda used contained chlorine, etc., instead of at once scorifying the p. p. pour dilute nitric acid on it, on the filter, to dissolve the silver arsenate and separate it from silver chloride which will remain on the filter. To the liquid add enough hydrochloric acid to precipitate all the silver in that as chloride, which collect on a filter, wash with hot water, dry and scorify with lead, adding a little soda.

The weight of the silver got multiplied by .2315 gives the weight of arsenic in the assay. Phosphorus and molybdenum may interfere in the process; these substances are not very common, but may occur in combination with lead in arsenical ores.

In treating ores by this method, especially if sulphuretted, or containing chromium, I prefer to proceed exactly as if preparing a solution for a wet assay of copper, up to the point at which iron and arsenic have been precipitated by ammonia, and, if needed, magnesia mixture; the p. p. is to be collected on a filter, washed, dried, and treated as above for arsenic, the ashes of the burned filter being added. The best way of burning the filter is to moisten it with solution of nitre, dry, and set fire to it in a dish. The above proceeding does not prevent the interference of phosphorus and molybdenum. Phosphorus may be got rid of by precipitating the arsenic from the solution by means of hydrogen sulphide; the p. p. must be collected and treated as in the other case; chromium will not interfere.

In precipitating arsenic by silver nitrate, the best way, though requiring more time, is to acidulate the alkaline solution with nitric acid, dilute largely if the substance contained sulphur, add a sufficient quantity of silver nitrate (which need not be neutral) then excess of ammonia so as to redissolve any p. p. and finally evaporate without boiling until the odor of ammonia is gone, when the silver arsenate will come down.

ASSAY OF ANTIMONY.

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*First Method.*—Fuse a weighed quantity of the ore with four parts of potassium cyanide in a small porcelain crucible in the muffle, or in an assay pot, according to quantity. Cool, and either break the pot or dissolve the slag by warm dilute hydrochloric acid; in either case clean the button with the latter, as it will usually not bear the hammer. If the ore contained little or no other reducible metal, the button may be weighed at once as metallic antimony; otherwise, as in presence of lead, etc., powder it, if brittle, laminate if tough, and boil in rather dilute nitric acid. Nearly all of the antimony will remain as a white powder, which, when washed with hot water, dried, and again fused with potassium cyanide, will yield a button of metallic antimony (and tin, if present, which is not probable).

To assay an alloy of antimony and tin, dissolve in aqua regia, with excess of hydrochloric acid; dilute the solution, adding more hydrochloric acid if a white p. p. forms, to redissolve that; place a piece of pure tin in the liquid; keep warm and maintain an excess of hydrochloric acid, collect, wash, dry, and weigh the precipitated antimony. The loss weight will be that of the tin in the alloy.

*Second Method.*—Fuse with five or six parts of a mix-

ture of sulphur and soda, equal parts, in a covered porcelain crucible; when fusion is complete and the excess of sulphur has volatilized, cool; extract with hot water, which dissolves the antimony. Filter; to the filtrate add gradually hydrochloric acid to acid reaction. Collect the p. p. on a filter; wash, transfer to a beaker, moisten the filter with solution of nitre; dry and burn it; add the ashes to the p. p. and treat the whole with red fuming nitric acid, or with a plenty of common nitric acid, which should not be heated so highly as to melt the sulphur, until all sulphur is oxidized. Evaporate nearly to dryness; cool, dilute, filter. The antimony remains on the filter, and may be determined as before directed.

*Third Method.*—Complex ores, or such as contain but a small percentage of antimony, may be dealt with as follows.

Proceed as for a wet lead assay as far at least as the first filtration, and, if lead is present, continue on that line until the lead is removed from the residue, using the ammonium acetate process, or the method with cold sodium carbonate solution.

The residue will contain the antimony, silica, and barium, and may now be sufficiently rich to be fused with potassium cyanide, or it may be digested with boiling hydrochloric acid, diluted a little but not enough to cause turbidity, filtered (best through asbestos or powdered glass) and washed with slightly diluted hydrochloric acid; the antimony will now be in the filtrate and may be recovered by precipitation as metal by means of iron or zinc, or the solution may

be boiled down with addition of nitric acid as long as that causes red fumes, and the white substance dried and reduced to metal by fusion with potassium cyanide.

ASSAY OF SULPHUR IN ORES.

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*First Method.*—Fuse ten grains or more of the powdered ore in a platinum or porcelain crucible, with about four parts of nitre and six parts of alkali carbonate, increasing the heat very gradually; cool; place the crucible, with its contents, in a suitable beaker or dish, cover with water, add a little more alkali carbonate, and boil gently until the matter is disintegrated and all that is soluble is dissolved. Filter, cool, add a drop or two of dilute hydrochloric acid and stir, but the liquid must remain alkaline; if a turbidity ensues (lead), you must filter again. Wash till the drops are neutral to test paper.

Acidulate the filtrate with hydrochloric acid, and evaporate to perfect dryness, breaking any clots that may form, by means of a glass rod. Moisten the mass with hydrochloric acid, add water, and digest warm. Filter, and again wash to neutrality. If the ore contained antimony, add some tartaric acid to the liquid. Heat, and add solution of barium chloride as long as it precipitates. Settle; collect the p. p. on a small, fine filter; wash with hot water, dry on the filter; transfer to a tared or counterpoised platinum or porcelain crucible, adding the ashes of the burned filter; heat to incipient redness, cool and weigh. The net weight of



p. p. multiplied by .1373 gives the weight of the sulphur.

*Second Method.*—Boil a weighed quantity of the powder and about an equal quantity of nitre or salt, in aqua regia, adding from time to time a crystal of potassium chlorate, if at hand, until all the sulphur is dissolved. Evaporate nearly to dryness; cool; add excess of strong solution of alkali carbonate, and boil gently for ten minutes at least. If lead is present, boil for thirty minutes; if strontium, boil for two or three hours, and longer still if the ore contained barium. The liquid must remain alkaline, more alkali carbonate being added if necessary to keep it so.

Filter; cool; add a few drops of dilute hydrochloric acid, and stir; filter again if necessary; acidulate the filtrate with hydrochloric acid, and proceed as directed in the first process except that you should evaporate to nearly dryness, then add hydrochloric acid, and dry completely.

In treating the ore with acid, if a part of the sulphur separates in melted drops, you may cool, remove, and wash the drops, adding the washings to the solution. Dry and weigh the drops of sulphur, proceeding with the solution as above. The weight of the sulphur thus got must be added to that found by calculation from the barium sulphate finally obtained.

Minerals may be assayed for that portion only of their sulphur that is available for various technical purposes; for the method, see other books.

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NOTE.—It may be as well to explain that the addition of a small quantity of acid to the alkaline solution, in both of the processes, is

for the purpose of developing some carbon dioxide to precipitate any lead which may have been dissolved in the boiling with alkali carbonate; hence, in the absence of lead, it is needless. To do this before filtering out the earthy carbonates might lead to the solution of a part of those which would then interfere. The addition of nitre or salt to the ore, in the treatment with acid, is for the purpose of securing the presence of a base for all sulphuric acid formed from the sulphur, in order to prevent its loss by evaporation. The addition of tartaric acid, when antimony is present, is to prevent the precipitation of any of that. About fifteen grains will suffice. The acid process is best for such ores as are likely to lose sulphur by heating.

## ASSAY OF SALT.

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IN buying salt for milling purposes it is often desirable to know its percentage of purity or its fineness.

Take 542 grain or gramme points=5.42 grains or 0.542 gramme;\* if it were pure it could convert 1,000 (=10 grains or 1 gramme) of silver into chloride. Dissolve in water; acidulate slightly with nitric acid; warm and add solution of silver nitrate; stir to curdling of the p. p.; settle and add a little more silver nitrate, and so as long as a p. p. is formed; an excess of silver nitrate will do no harm. Settle; filter, and wash with hot water until the drippings cause no cloud in salt solution. Dry the p. p. and dress on filter with litharge, soda, and borax mixed. Put filter and all into a crucible; smelt and cupel as in an ore assay. Weigh the resulting silver bead, adding a correction for cupellation loss according to experience. The bead weight in points is the fineness of the sample, and that divided by 10 is the percentage of pure salt (or the equivalent of that for utility).

Marsh salt often contains soda or lime, and if used

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\* In order to get a fair average, it will be best to take ten or one hundred times the quantity named, dissolve in water; filter; weigh or measure the solution, then, by weight or measure, take one-tenth or one hundredth part of the liquid for the assay.

in conjunction with bluestone or acid, as in working silver ores by amalgamation, renders a portion of these useless. In acidulating the solution as above, observe if much effervescence ensues, as it may be more profitable to pay a higher price for a better article.

To find how much bluestone the impure salt will counteract, take 100 parts (say grains) of the sample, dissolve in water and add 10 parts of bluestone also dissolved in water. Boil for a few minutes, and if the liquid is discolored add another quantity of bluestone and again boil. Filter and wash; acidulate the liquid with sulphuric or hydrochloric acid; precipitate, wash, and weigh the copper remaining in solution, exactly as in making a copper assay (or determine the copper by either of the other processes given). The weight of the copper multiplied by 4.06 is the weight of bluestone not made useless by the impurities in the sample, and this deducted from the total weight of bluestone used leaves the weight made useless by 100 of the sample. The blue crystals of bluestone should be used, rejecting any white portion.

To find out how much of the acid in use the impure salt will neutralize, take 100 or 1,000 grains of the sample, dissolve in water and heat the solution. Take also a weighed quantity of the acid, say 10 or 100 grains, and pour it into 5 or 6 times as much water,\* mix, and weigh again, or measure the mixture, if more convenient. Add the acid liquid, little by little, to the salt solution, stirring, and, when effervescence ceases,

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\* Never pour water into strong acid, but pour the acid into the water to avoid explosion,

testing after each addition with blued litmus paper until that is slightly reddened. The proportion of the dilute acid used indicates the proportion of the acid taken which was necessary to counteract the alkaline matter in the taken weight of the sample.

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NOTE.—In all those processes in which the substance got is different from that sought, the practise has been to state that the former contains so much per cent of the latter, or the weight got must be multiplied by some number, which is often an inconvenient one, in order to ascertain how much of the substance sought was present in the assay.

It is much simpler to take such a weight of the assay substance as would, if it consisted wholly of the substance sought, give 100, by similar weight, of the substance to be got and weighed. The weight obtained would then be the percentage of the substance sought which the assay contained. In the wet assay of lead, for example, when not the lead itself but the sulphate is to be weighed, as lead sulphate contains 68.32 per cent of lead, it is easily seen that 68.32 parts of pure lead would give 100 parts of sulphate, and 68.32 parts of a substance which is only partly lead would give so much sulphate as would correspond to the percentage of lead in it.

In case the quantities should be inconveniently large or small, in the system of weights used, we may take a simple multiple or an aliquot part of the whole, and divide or multiply the weight got accordingly by some simple number.

Following are the weights of ore, etc., to be taken on this system to be assayed for the respective substances weighed in the forms named. The weights may be grains, hundredths of grains, grammes, milligrammes, etc., as may be convenient; the weight of the substance got, stated in similar terms, is the percentage of the substance sought.

68.3 for lead as sulphate.

36.5 for zinc as ammonio-phosphate.

79.85 for copper as oxide.

34.85 for copper as xanthate.

19.6 for nickel and cobalt as xanthate.

13.8 for sulphur as barium sulphate.

23.15 for arsenic as metallic silver by substitution.

Whenever an assay of any kind is undertaken, a record, or at least a memorandum, should be made of the quantity operated on, and any other particulars which require to be kept in sight. In cases of rather complex operations it is a good plan to write down the entire proposed course of treatment, and to check off each step as it is performed, especially should this be done when variations from the prescribed course are contemplated, as will often occur if the workman is intelligent, and has some knowledge of chemistry.

When a multiple or aliquot part of a standard quantity is taken, the fact should appear on the record; for example, in the copper assay where the copper is to be weighed as xanthate, 34.85 grains would be too much of a rich ore, giving a too great volume of p. p. for rapid working. A tenth of the quantity will suffice in such a case, and may be written  $3\frac{4}{10}^{.85}$  grains, in

order to show that the weight of copper xanthate got in grains is to be multiplied by 10 for the percentage. On the other hand, if gramme weights were used, 35.85 grammes would be too much of any ore, and 35 milligrammes would be too little of any; in this case 349 milligrammes might answer. A little practise will give facility in this system of weighing.

In the assay of manganese for chlorination works, the substance sought is manganese dioxide; the substance got (weighed) is carbon dioxide, and 99 of the former are indicated by 100 of the latter, hence we take 99 of the assay substance. In the arsenic assay, as here given, arsenic is sought and silver is got; 100 of silver in this case indicate 23.15 of arsenic, and we take 23.15 or  $2\frac{3}{10}$  grains of the ore. In the copper assay, if we are going to precipitate the copper in the metallic state, copper is sought and copper is got, and copper of course contains 100 per cent of copper, so we take 100 of ore in any convenient term of weights, or we take  $1\frac{00}{100}$  perhaps; but if we are going to get the copper in the form of oxide, containing 79.85 per cent of copper, we take 79.85 of ore or  $7\frac{985}{100}$ , etc., according to the estimated richness of the ore and the denomination of the weight.

The system is not advantageous when two or more substances are to be determined from one assay quantity, unless all are to be weighed in forms which have equal percentage values. This is the case with nickel and cobalt when weighed as xanthates.

A similar device is applicable to volumetrical assays, and by means of it we may so arrange as to read

the result of an assay, in percentage, directly from the burette.

To do this we make the determining solution of such strength as that 100 c. c. of it shall correspond with a convenient weight of the metal, etc., to be determined, and we use that weight of substance for the assay. The reading of the burette then gives the percentage of the metal sought which the substance assayed contains. The strength of the solution and the weight of substance to be used are deduced from the combining weights of the elements involved in the reaction.

But, however accurately a solution may be standardized, the titre will not remain constant, owing to evaporation, to chemical changes, or to expansion and contraction due to changes of temperature; hence the need of making check assays on substance of known composition. The correction for aberration of titre may be made in either of two ways. First, by calculation, as follows: Let  $n$  = the result of an assay on the standard quantity of substance, as got by a solution which varies from its proper standard, and let  $t$  = the actual titre of the solution as found by the check: then,  $n \times 100 \div t = x$  = the true result. *Example.*—An assay for a metal is made with a solution of which the actual titre, or  $t$ , is 98.2 c. c., instead of 100 as it should be; the apparent result, or  $n$ , = 23.7 per cent, then,  $23.7 \times 100 \div 98.2 = 24.13$  per cent, the true result.

The second method is to change the weight of substance taken for the assay, so that it may correspond with 100 c. c. of the solution, as follows: Let  $n$  = the



standard weight of substance, and let  $t$  = the actual titre of the solution, then,  $n \times 100 \div t = x$  = the weight to be taken. *Example.*—The standard weight of substance, or  $n$ , is 0.686 gramme, and the actual titre of the solution,  $t$ , is 98 c. c.; then  $0.686 \times 100 \div 98 = 0.7$  gramme of substance to be taken for the assay so that the reading of the burette may give the percentage correctly. This latter method is the best when a number of samples have to be assayed for the same metal, because the one calculation answers for all. Thus also a weight may be found which will agree with any desired strength of solution, or, conversely, a solution may be made of a strength suited to any desired quantity of substance, by trial and modification, so that a knowledge of the chemical reaction involved is not indispensable.

Some rather fascinating methods in volumetrical assaying of metals, etc., are based on the reaction between iodine and sodium thiosulphate (erroneously called hypo-sulphite), in which the iodine combines as sodium iodide and the sodium thiosulphate is converted to tetrathionate, so that either of the original substances, the quantity of which is known, becomes the means of measuring an unknown quantity of the other, and as several metal salts, such as cupric sulphate possess the property of setting iodine free from potassium iodide in equivalent proportion, the measurement of the iodine thus set free gives the measure of the quantity of metal salt which set it free, and therefore of the metallic base of that salt.

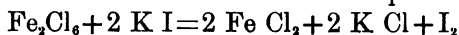
In some cases, as for instance that of arsenic, the

reverse of this occurs, that is, free iodine, if present, goes into combination, hence, if a known quantity of free iodine is added, a part of it becomes not free, and the remaining free portion, being measured by means of a standard solution of sodium thiosulphate, indicates the quantity which has combined, which, in its turn, denotes the quantity of arsenic present. This appears rather complicated, but is not necessarily so in practise.

An obstacle to the more general use of these elegant methods seems to be that, as they are described in the books, either a good deal of calculating must be done or a special solution of thiosulphate must be made and kept at hand for each kind of assay, one for copper, another for iron, etc. Many assayers lack time or inclination, a few, perhaps, the ability to go behind the formal prescriptions of the books.

It is not my present purpose to describe these methods of assaying; all necessary instructions for preparing the solutions and making the titrations can be found in "Thorp's Analysis," "Beringer's Handbook of Assaying," and other works. My object is to show how a single solution of sodium thiosulphate may conveniently be used for the assaying of a variety of substances, and the percentage of the metal, etc., determined without calculation, or with a minimum of figures.

In the assay of iron, by this method, the reaction by which iodine is set free is thus expressed:—



This means that 56 parts of iron, in suitable solution

set free 126.85 parts of iodine, hence 1 gramme of iodine thus set free indicates the presence of 0.44 466 gramme of iron. One gramme of iodine is equivalent to 1.958 grammes of crystal sodium thiosulphate, hence the latter is equivalent to the above quantity of iron.

Now it is clear that, if we use, for an assay, 0.4415 gramme of substance which is 100 per cent iron, the assay will consume 1.958 grammes of thiosulphate, and if that quantity is contained in 100 c. c. of our thiosolution, the reading of the burette will be 100 c. c. Now, suppose the substance to be half iron and half something else, clearly the assay will consume 50 c. c. of solution, and the reading of the burette will again indicate the percentage of iron, and so with any proportion. It will perhaps be better to use double the above quantity of substance, and to make the thiosulphate solution twice as strong, that is, containing 3.916 grammes in 100 c. c. or 39.16 to the litre; the percentage will be read from the burette as before.

Assuming that the solution of thiosulphate contains 39.16 grammes of the crystals to the litre, the weights of substance to be taken for the different assays are:—  
For iodine.....2.0000 grammes.

" copper .....	0.9996	"	
" iron .....	0.8829	"	
" antimony .....	0.9460	"	
" manganese dioxide...	0.6860	"	by distillation with H Cl, etc.
" potassium dichromate	0.7757	}	by digestion with H Cl.
" potassium chlorate...	0.2958		
" potassium cyanide*	0.5133	"	

For tin\*.....0.9302 grammes.

“ arsenic\*(direct met'od)0.5912 “

“ zinc\*.....0.5128 “ convert to sulphide and treat with H Cl developing H<sub>2</sub> S, etc.

The above weights are *n* for the respective substances with the given solution, but if a check assay on a known quantity of iron, copper, etc., shows the standard to have changed, the weight may be adjusted to it by the rule given above. The reading of the burette will then be the percentage of the substance sought in the sample, except as to those marked \*, which are of the class in which iodine is taken into combination, and the reading of the burette indicates the uncombined portion.

In that class of assays in which iodine is absorbed the practise is to add a known quantity of iodine, in excess, and determine the uncombined residue by means of the thiosulphate solution. The most convenient way in which to apply the iodine is in the form of a standard solution, and I think the best way of working the assays (of solids) is to take a quantity of substance corresponding to the titre of the thiosulphate solution, as explained above, prepare the assay as directed in the books, add a quantity of iodine solution equivalent to 100 c. c. of the thiosulphate solution, and then titrate. The number of c. c. of thiosulphate solution consumed, subtracted from 100, leaves the percentage sought.

For a thiosulphate solution containing 39.16 grammes to the litre, as described, the equivalent iodine solution will contain 20 grammes to the litre, but, owing to the probable deterioration of the former, it will be necessary to determine how much of the iodine solution is actually equivalent to a given volume of it. Take 100 c. c. of the thiosulphate solution, add starch paste, and run in the iodine solution from a burette until a permanent blue tint appears; the quantity of iodine solution consumed is that which is equivalent to 100 c. c. of the other. It may be desirable to make the iodine solution stronger, so as to limit the quantity of liquid.

It is scarcely needful to remark that in the case of assays of substances which are poor in the metal sought, multiples of the quantities may be taken, and treated as the single quantity, the result being divided accordingly.

In case we wish to determine several of the components of a substance, we may either make a separate assay for each, or we may take that weight of substance which is requisite for the component requiring the greatest quantity, and proceed in a manner which is best illustrated by an example: Suppose we wish to assay an ore for copper, iron, antimony and arsenic, we take the proper weight for copper, that is  $\frac{0.9996 \times 100}{t}$  gramme, dissolve it and separate the

metals required by appropriate methods, dissolve each in a suitable manner for the assay, determine the whole of the copper, add to each of the other solutions,

in a tared beaker, water to make up  $\frac{99.96 \times 100}{t}$

grammes of solution; from the iron solution remove so much as to leave  $\frac{88.3 \times 100}{t}$  grammes, and determine

the iron; from the antimony solution remove so much as to leave  $\frac{94.6 \times 100}{t}$  grammes and determine the anti-

mony, and, in like manner, take  $\frac{59.12 \times 100}{t}$  grammes

of the arsenic solution for the assay of arsenic. In each case, except the last, the percentage will be read directly from the burette, the percentage of arsenic will be the difference between the burette reading and 100. The same thiosulphate solution will be used for all.

I think the number of substances which may be assayed by means of iodine and sodium thiosulphate might be extended considerably. I have suggested one, namely, zinc, which I have nowhere seen mentioned in this connection, but which can be determined on this principle, either through the direct effect of hydrogen sulphide on iodine, or through its action on ferric chloride. Any substance which can be caused, directly or otherwise, to set iodine free, or to combine it, in equivalent proportion, or which can decompose the acid of a thiosulphate, is theoretically amenable to the system.

It is always allowable, and often desirable, not to weigh out any exact stated quantity of substance, but to take an approximate quantity, weigh it, note the weight, proceed with the assay, and calculate by proportion the result which the proper quantity of substance would have given. Before relying implicitly on any new method, it is well to check by an older and more familiar one, in order to guard against errors, either in the directions, through misprints or otherwise, or in the attempt to follow the direction.

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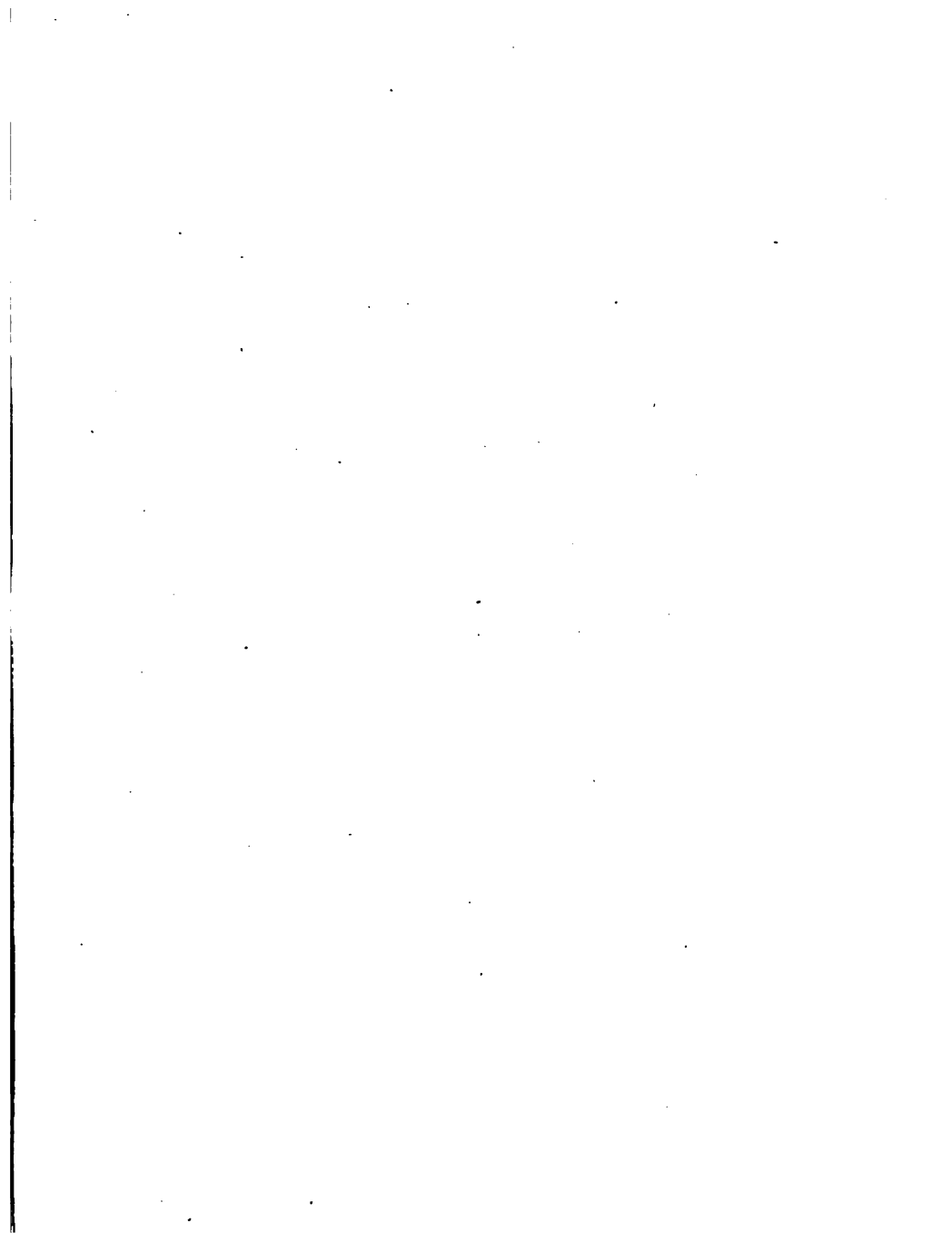
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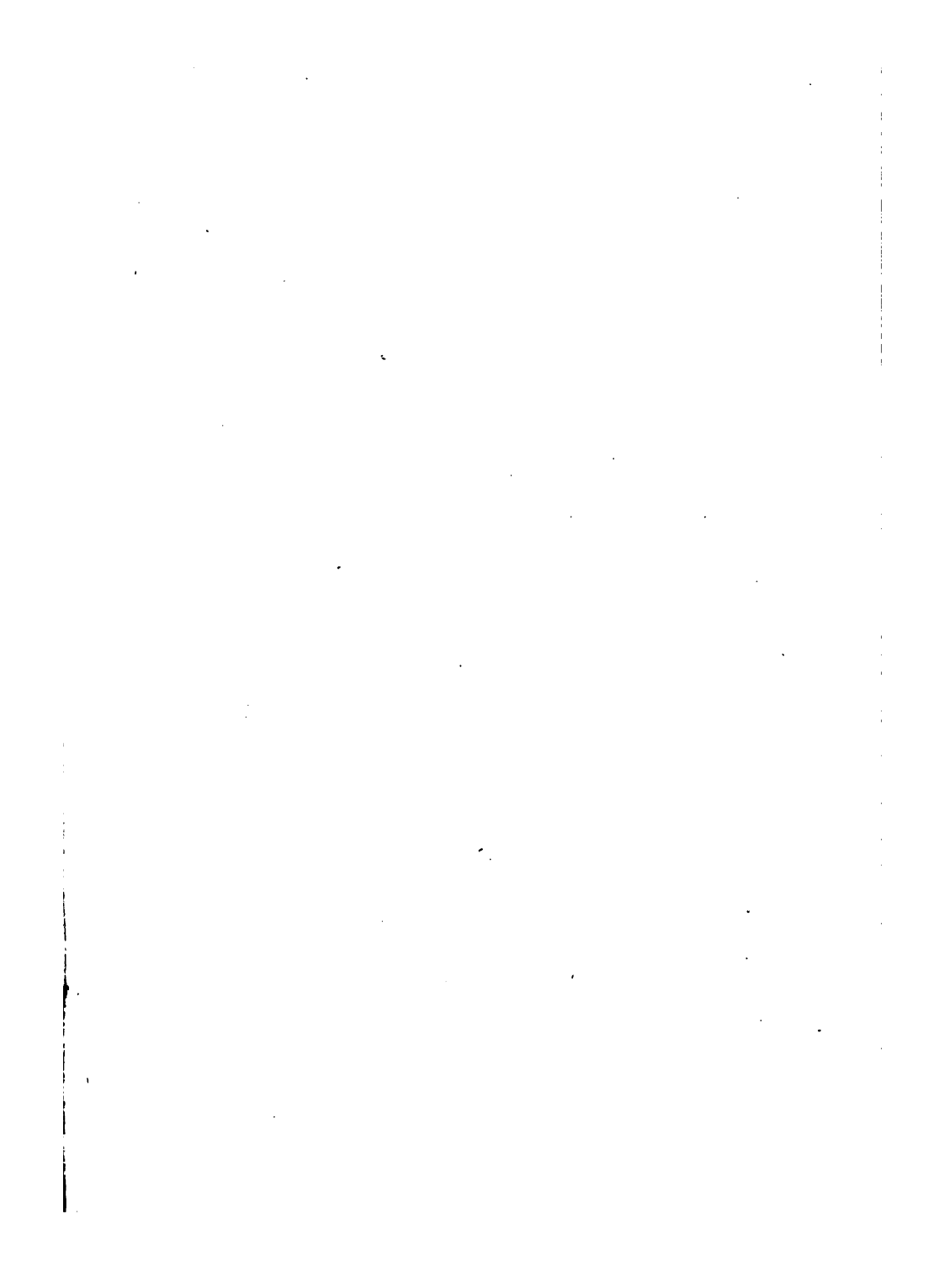












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